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DEVELOPMENT OF A WATER SOLUBLE FOAM PACKAGING MATERIAL

T. J. McCown, et al

McDonnell Douglas Astronautics Company

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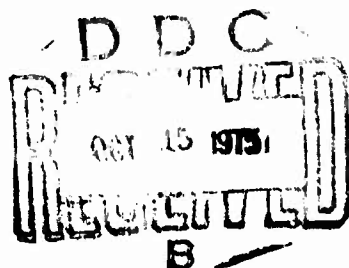
Army Natick Development Center

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TECHNICAL REPORT
75-88-FE

DEVELOPMENT OF
A WATER SOLUBLE FOAM PACKAGING MATERIAL

McDonnell Douglas Astronautics Company

St. Louis, Missouri 63166

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KLUCEL	SOLUBILITY (WATER)	EXPOSURE
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DEVELOPMENT	FOAM	FRESH WATER
FEASIBILITY STUDIES	PACKAGING FOAM	SALT WATER
CELLULOSIC RESINS	PACKAGING MATERIALS	DISSOLUTION

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The purpose of this program was to investigate the feasibility of developing and reliably reproducing a water soluble foam from hydroxypropyl cellulose resin (Klucel) and to establish product parameters.

The program was divided into two phases. In Phase I, the feasibility of extruding the water soluble thermoplastic resin into a candidate packaging material was successfully demonstrated. Hydroxypropyl cellulose was extruded

by conventional means into a loose-fill packaging foam having a compressive stiffness conforming to Federal Sepcification PPP-C-850, Cushioning Material, Polystyrene Expanded, Resilient, Class 4 (Extra Firm). In Phase II, the loose-fill foam product produced from this resin was tested using the methods detailed in Federal Specification PPP-C-1683, Cushioning Material, Expanded Polystyrene, Loose-Fill Bulk and standard properties were established. Additional investigations conducted on the loose-fill samples were:

   o Effects of long and short term relative humidity exposure.
   o Solubility and dissolution rates in fresh and salt water.

Long term humidity exposure caused the material to deteriorate and lose all foam cushioning properties.

Complete dissolution of the Klucel foam floating and submerged in fresh and prepared sea water was demonstrated.

FOREWORD

As a result of extensive use of foam plastics for cushioning materials
by the various DOD agencies, disposal of the material after use is an
ecological problem, particularly at depots and aboard ships at sea. A
water soluble, nontoxic cushioning material, hydroxypropyl cellulose,
was proposed by McDonnell Douglas Corporation as a solution to this problem.
Subsequently, the US Army Natick Development Center contracted with
McDonnell Douglas to determine whether hydroxypropyl cellulose could
be reliably fabricated into a foamed product using conventional foam-
ing processes; and to establish the standard product parameters on the
material produced.

The project was carried out by the McDonnell Douglas Astronautics
Company – East, Saint Louis, Missouri. The Program Manager was Mr.
Thomas J. McCown and the Principal Investigator was Mr. Gilbert E.
Wahlmann. The extrusion process work was done under subcontract by
Mr. Donald Murray of Gloucester, Massachusetts. Project Officer for
the US Army Natick Development Center was Mr. Raymond T. Mansur of the
Food Packaging Division, Food Engineering Laboratory and the Alternate
Project Officer was Mr. Charles F. Macy of the Pollution Abatement
Division, Food Sciences Laboratory.

TABLE OF CONTENTS

FIGURES

FIGURES (Continued)

TABLES

Development of a Water Soluble Foam Packaging Material

1.   INTRODUCTION

1.1   Background

Current packaging techniques for ordnance, hardware, electronics and other supplies used by the various Department of Defense agencies employ plastic foams extensively for foam-in-place, loose-fill, and preformed shape protective materials.

Although plastic foams provide lightweight, low cost packaging materials, disposing of the foam after uncrating and unpacking can be an ecological problem. Consequently, methods are needed to effectively dispose of the discarded packaging materials without causing an adverse effect on the environment. A potential solution to this disposal problem would be to develop a packaging material which dissolves in water without leaving undesirable by-products. Hydroxypropyl cellulose resin (Klucel*), which has been used in foodstuffs for many years, is a candidate material which might be used for such a product application. This resin is water soluble, nontoxic, and has an inherently low equilibrium moisture content. Foamed products have previously been molded from various types of hydroxypropyl cellulose but the manufacturing feasibility had not been determined.

1.2   Purpose

The purpose of this project was to determine the feasibility of producing a water soluble packaging foam from hydroxypropyl cellulose resin and to establish standard product parameters on the material produced.

1.3   Objectives

The objectives were:

o Evaluate conventional methods for producing a packaging foam material using hydroxypropyl cellulose resin.
o Establish manufacturing reliability.
o Produce representative samples of hydroxypropyl cellulose foam to comply with Class 3 (Firm) or Class 4 (Extra Firm) of Federal Specification PPP-C-850D; Cushioning material, Polystyrene Expanded, Resilient
o Test to establish standard properties for hydroxypropyl cellulose foam material including effects of humidity and water solubility characteristics.
o Provide samples of finished product.

*Registered trademark of Hercules, Inc.

6

1.4 Organization

The project was divided into two phases:

o   Phase I - Feasibility Demonstration
o   Phase II - Property Determination

2. PHASE I - FEASIBILITY DEMONSTRATION

The feasibility to produce a water soluble, loose-fill foam packaging
material was demonstrated in the following manner.

2.1 Resin Material

The basic resin used to make the water soluble foam product
(Figure 1) was a hydroxypropyl cellulose manufactured by Hercules,
Incorporated, called Klucel. This resin is prepared by reacting alkali
cellulose with propylene oxide at elevated temperatures and pressures.
The propylene oxide can be substituted through an ether linkage at the
three reactive hydroxyls present on each anhydroglucose monomer unit of
the cellulose chain (Reference 1). Published information suggests that
the etherification takes place in such a way that the hydroxypropyl
substituent groups contain almost entirely secondary hydroxyls. The
secondary hydroxyl present in the side chain is available for further
reaction with the oxide and chaining out may take place.

Since hydroxypropyl cellulose is water soluble, the effect of rela-
tive humidity on packaging stability is important. Despite being
readily soluble in water, items foamed from hydroxypropyl cellulose are
nontacky and do not readily fingerprint during handling (Reference 2).
Equilibrium moisture, or the maximum amount of moisture which the
material will absorb at a given relative humidity, is the key to this
performance. At 73°F and 50 percent relative humidity, hydroxypropyl
cellulose has an equilibrium moisture content of four percent. It
should be noted that hydroxypropyl cellulose is far less hygroscopic
than other water soluble polymers.

With regard to toxicity, hydroxypropyl cellulose is physiologically
inert. The results of repeat insult patch tests on humans have not
disclosed any evidence that it is either a primary skin irritant or a
skin sensitizing agent (Reference 3). This resin is nonnutrient, being
nutritionally equivalent to purified cellulose (Reference 4).

2.2 Processing Methods

The extrusion process was selected as the conventional fabricating
technique to demonstrate the feasibility of producing a loose-fill foam
material from hydroxypropyl cellulose resin. Pilot plant and laboratory
size extruders normally used to manufacture polystyrene, polyethylene and
polypropylene foams were employed to produce the Klucel foam samples. In
general, the equipment melts and mixes the resin formulation, provides access
for injection of the foaming agent, and forces the product through the ex-
truder die to form the desired shape.

1. A. J. Desmarais, "Hydroxyalkyl Derivaties of Cellulose", Industrial
   Gums, 2nd Edition (1973), pg. 650.

2. Ibid, pg 665.

3. Ibid, pg. 666.

4. Hercules, Incorporated, "Klucel-Hydroxypropyl Cellulose; Chemical
   and Physical Properties", Brochure (1971), pg. 4.

FIGURE 1 - WATER SOLUBLE FOAM LOOSE-FILL PACKAGING MATERIAL

2.3 Formulation (Table 1)

The material and processing characteristics of hydroxypropyl cellulose resin were examined and beginning formulations were determined In addition to the resin, ingredients included an anticake agent, plasticizer, lubricant, stabilizers, nucleating agent and foaming component.

2.3.1 Hydroxypropyl Cellulose (Resin) - As stipulated by contract, Klucel was used as the thermoplastic resin. Klucel is available in several grades which vary in molecular weight (M_W) from 50,000 to 1,250,000 (Reference 5). Grade J (M_W = 150,000) was chosen from other Klucel grades because it has the best flow characteristics, which is beneficial in an extrusion process. A change to a higher molecular weight grade of Klucel results in a softer polymer but reduces the melt flow characteristics (Reference 6). A decrease in polymer molecular weight increases both the product stiffness and melt flow

2.3.2 Silicon Dioxide (Anticake Agent) - Compression of the resin by its own weight causes compaction and fusion into a lumpy solid To prevent this, a small quantity of silicon dioxide was included in the formulation to serve as an anticaking agent.

2.3.3 Polyethylene Glycol (Plasticizer) - To impart flexibility and workability into the plastic melt and to reduce the fibrous behavior and delamination tendency of the thermoplastic resin, a low molecular weight (400 $\pm$20), water soluble polymer of ethylene oxide was used as a plasticizer. It has good heat stability and is not a skin irritant.

In the initial runs, propylene glycol was used as the plasticizer but was found to be less suitable due to its low boiling point and tendency to flash off at the die.

2.3.4 Polyethylene Glycol (Lubricant) - A high molecular weight (3350 $\pm$350), water soluble polymer of ethylene oxide was used as a lubricant. A waxy solid, this material was added to provide good anti-stick and antiblocking properties to the formulation during its passage through the extruder die. This was a replacement lubricant for glycerol monostearate which tended to exude to the surface of the product and produce a waxy or sticky feel.

2.3.5 Butylated Hydroxytoluene (BHT) - Hydroxypropyl cellulose resin is a cellulose ether whose molecular weight can be significantly reduced by cleavage of glucose units when severe conditions of heat and oxidation prevail. Without the BHT to act as a stabilizer, the molecular weight would be degraded and constant variations in the melt viscosity of the formulation would result, producing erratic behavior during extrusion.

2.3.6 Laurylthiodiproρionate (LTDP) - LTDP functions as a stabilizer by decomposing peroxides to make them less effective as degradation agents.

2.3.7 Magnesium Silicate (Talc) - Ultra fine (17 m^2 surface area/g) and highly pure (98 percent) magnesium silicate powder was added as a processing aid and as a nucleating agent to provide sites for the formation of bubbles in the same manner as a cloud is seeded to produce rain. Only enough talc was used to lightly dust the outside of the formulation pellet. Talc has also been used effectively with polystyrene and polyethylene foaming operations to produce a smooth, flat surface effect.

2.3.8 Freon 11/Freon 12 (Foaming Agent) - The foaming agent used throughout this investigation was duPont Freon*. It was chosen because it is one of the easiest, safest and most efficient materials available for foaming operations. Also, it has the ability to produce fine cell structure in polystyrene and polyethylene foam products. Freon 12 was very satisfactory but it was found that a 70% Freon 12 and 30% Freon 11 blend minimized occasional popping at the die and was used to produce the Phase II test specimens.

2.4 Equipment and Process Description

2.4.1 Blender - The nucleating agent (talc) was blended with the rest of the formulation ingredients, previously pelletized into one-eighth inch diameter spheres, in a small cement mixer type blender. Blending was continued for approximately ten minutes to provide a dust coating over the pellet exterior. The blended batch was then loaded by hand into the feed hopper of the extruder.

2.4.2 Extruder - Foaming feasibility was determined using a two-stage Extro extrusion press manufactured by Gloucester Engineering Company of Gloucester, Massachusetts. The extruder features tandem screws in series with one another (Stage 1 and Stage 2). A schematic of the floor plan for the extruder complex is shown in Figure 2. The circled figure numbers indicate the location and direction of the figures which follow.

The ingredients are delivered into the barrel of the primary extruder, Stage 1 (Figures 3 and 4), which contains a 2-1/2 inch diameter blending screw. The barrel housing has five heater stages and the ingredients are mixed and heated until a uniform molten state is attained. Near the end of the barrel, the Freon liquid foaming agent is injected into the molten plastic under pressure supplied by the pumping units (Figure 5) and controlled by settings on the control panel (Figure 6).

The two extruder stages are joined by a coupling (Figure 7). The temperature of the plastic melt is continually monitored at this interface location.
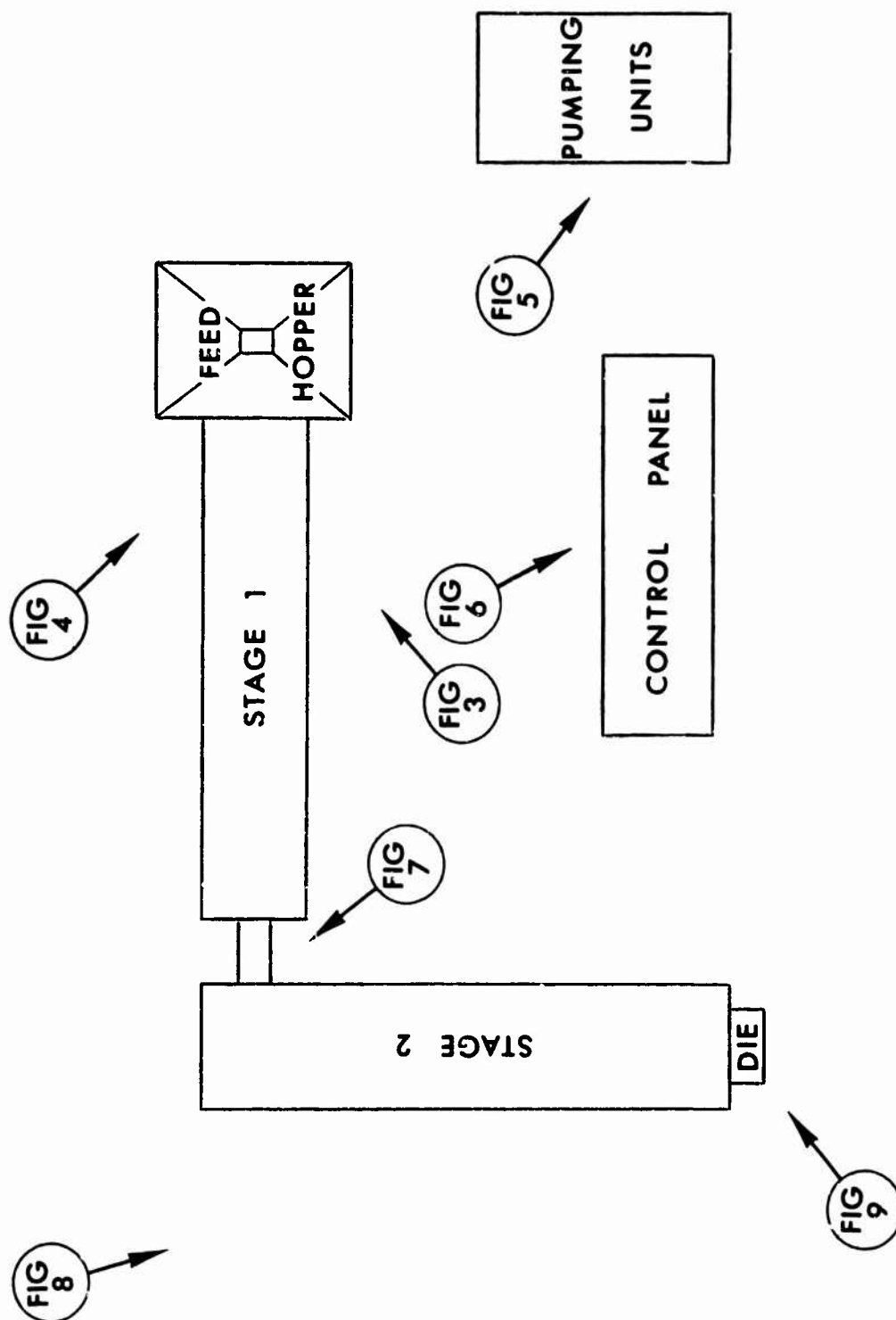
*Registered trademark of duPont.

11

FIGURE 2 - TWO-STAGE PILOT PRODUCTION EXTRUDER USED FOR PHASE I DEVELOPMENT WORK
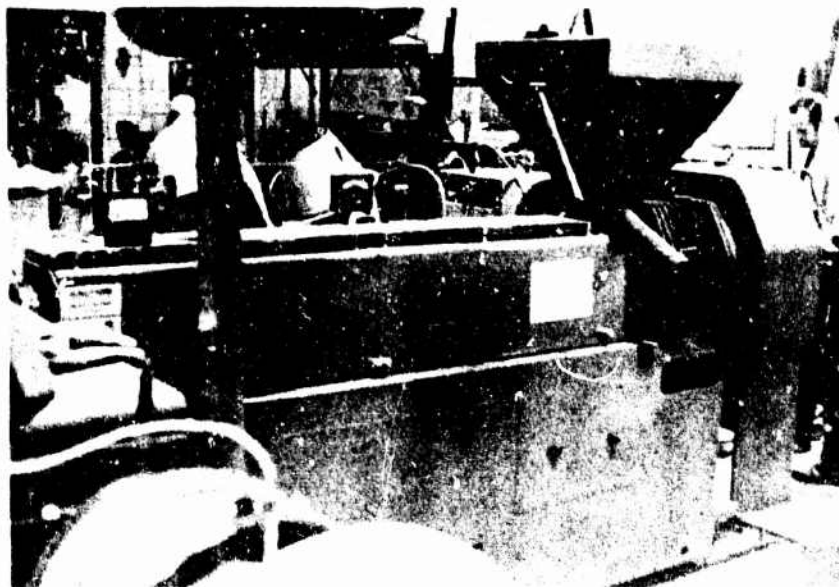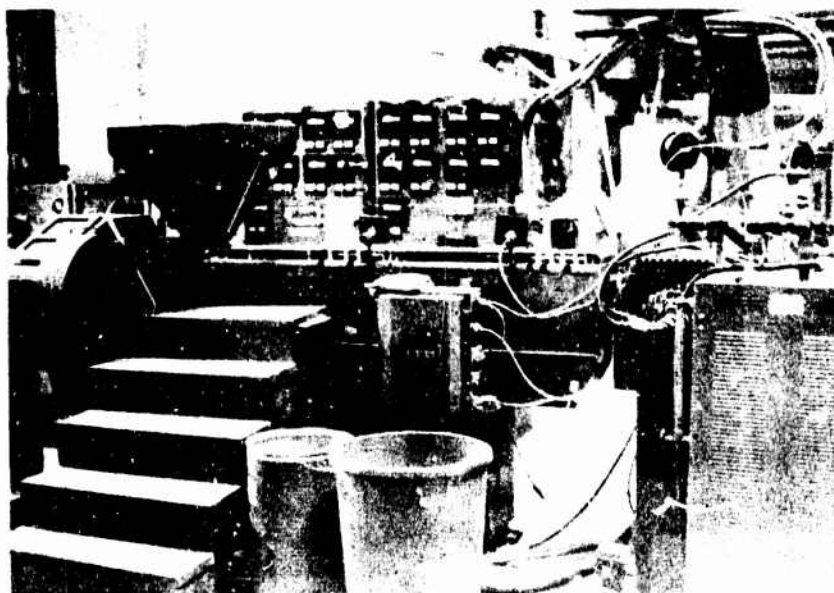
FIGURE 3 -- EXTRUDER STAGE 1 AND FEED HOPPER

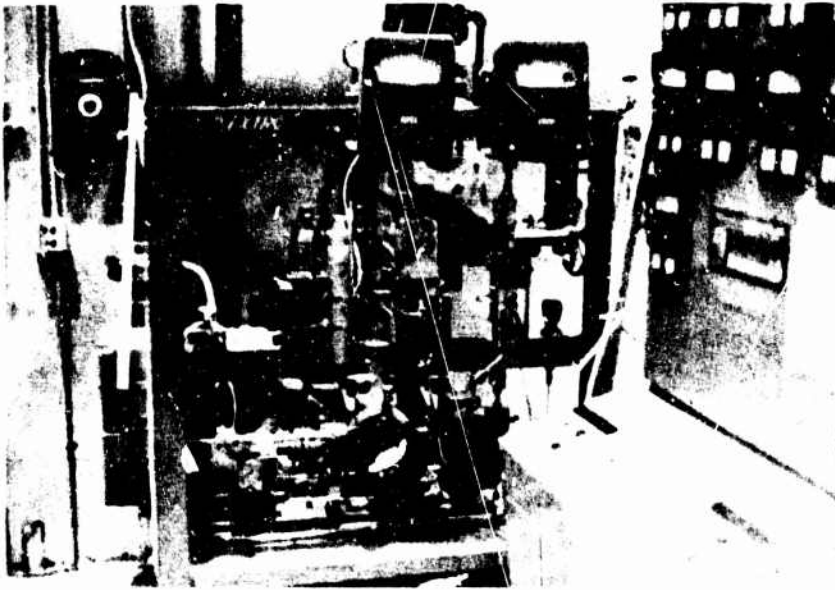FIGURE 4 - EXTRUDER STAGE 1 (REAR) AND FEED HOPPER

FIGURE 5 - PUMPING UNITS
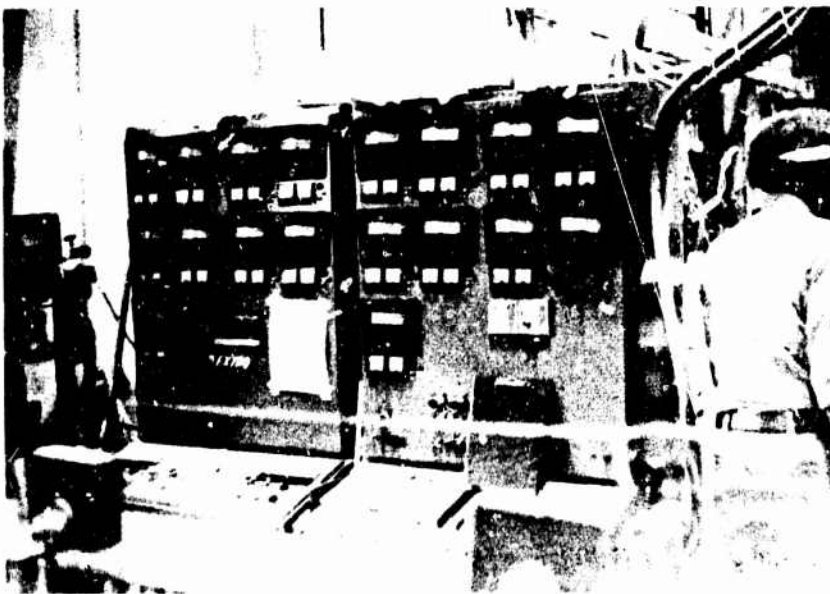
FIGURE 6 - CONTROL PANEL

14
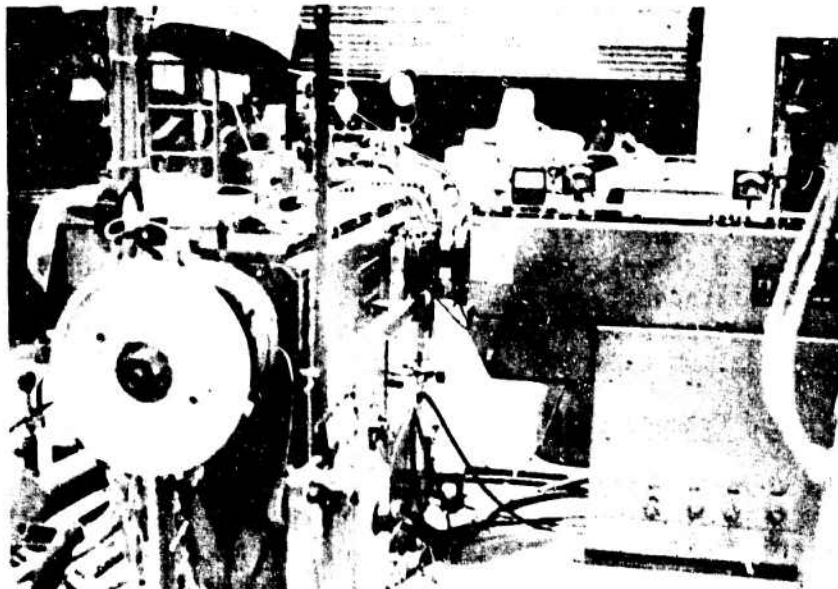
FIGURE 7 - COUPLING BETWEEN EXTRUDER STAGES
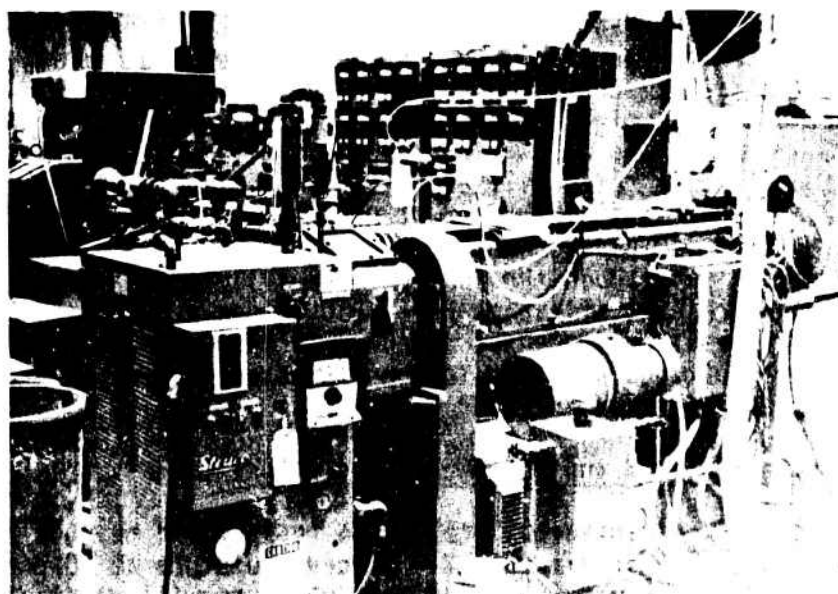
FIGURE 8 - EXTRUDER STAGE 2 (REAR)

The second extruder stage (Figure 8) is used for cooling the melt. In the barrel of this stage, another 2-1/2 inch diameter screw is used to distribute a uniform temperature throughout the plastic melt while the temperature is being lowered to increase the discharge pressure at the die and facilitate the foaming process. Four temperature zones are controlled in this stage and a pressure measurement can be made in the die adapter just before the material reaches the extrusion die. A 1/8 inch diameter die opening (Figure 9) was used for the Phase II test evaluation and production sample material.

During the latter part of the Phase I feasibility study, a scaled-down version of the Extro extruder (Mini Extro) was used. The screw size of this extruder was 1-1/4 and 1-1/2 inches diameter for the Stage 1 and Stage 2 screws, respectively, and it was operated at its maximum throughput capacity of approximately 24 pounds per hour. The Extro extruder was operated at approximately 40 percent of its 150 pound per hour maximum capacity.

2.5 Material and Process Development

During the course of this investigation it became apparent that certain processing conditions must be present in critical combinations if a foam material with the required physical and mechanical properties was to be produced and reproduced reliably. These are:

- o Mixing ratio
- o Barrel temperature
- o Barrel pressure
- o Extrusion rate.

All are interrelated. Changing one will modify each of the others and variations in material densities, cell structure, cell uniformity, surface conditions and mechanical properties will result. In addition, altering the molecular weight of the resin, the amount of the foaming agent or the amount of the nucleating agent as well as the moisture content of the starting resin will also contribute to modifications in the end product. The final foam product resulted from a series of adjustments to both formulation and processing parameters. The final formulation chosen to produce the foamed loose-fill samples, which complied with Federal Specification PPP-C-850, Class 4 (Extra Firm) compressive stiffness requirements, is shown in Table 1. Typical extruder control settings utilized in the extrusion process to produce the above samples are shown in Table 2.

2.6 Problems Encountered and Recommended Solutions

2.6.1 Moisture - Hydroxypropyl cellulose resin which has not been predried will contain approximately 3-4 percent moisture at 50 percent relative humidity conditions. Some of this moisture will vaporize into steam in the extruder barrel and vent back to the feed hopper where it condenses, causing entering feed material to agglomerate and choke the
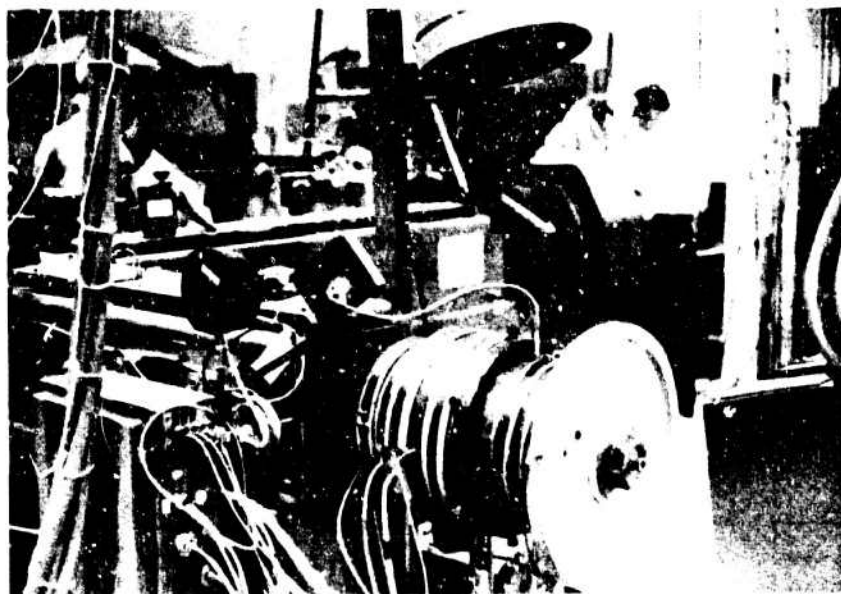
FIGURE 9 - EXTRUSION DIE

17

TABLE 1 -- LOOSE-FILL FORMULATION

INGREDIENT	FUNCTION	SOURCE	PERCENT BY WEIGHT
Hydroxypropyl Cellulose	Resin, Predried	Hercules, Inc. Klucel, Grade J	98.1
Silicon Dioxide*	Anticake Agent	Degussa, Inc., Pigments Div. Aerosil	0.2
Polyethylene Glycol	Plasticizer	Union Carbide Corp. Chemicals & Plastics Div. Carbowax 400	0.5
Polyethylene Glycol	Lubricant	Union Carbide Corp. Chemicals & Plastics Div. Carbowax 4000	0.5
Butylated Hydroxytoluene (BHT)	Stabilizer, Antioxidant	Shell Chemical Co. Ionol CP	0.2
Laurylthiodipropionate (LTDP)	Stabilizer, Peroxide Decomposer	American Cynamid Co. Plastic Additives Div. Cyanox	0.2
Magnesium Silicate** (Talc)	Nucleating Agent	United Sierra Division Cyprus Mines Corp. Mistron Vapor	0.3

* Normally supplied combined with resin.

** Added as separate ingredient to pelletized formulation.

18

flow. The material which goes through the extruder also contains some steam which causes surging and popping as the mixture is forced through the die. The resulting material is also very soft and has a tendency to collapse as the water contained serves as additional plasticizer. Higher humidity operating conditions were found to aggravate this problem as the feed material tends to stick to the sides of the feed hopper.

An attempt was made to remove this moisture from the resin in the first stage barrel during extrusion by a process called starve feeding. In this method, sometimes used successfully with other plastic resins, the feed rate to the extruder is decreased to create a void space in the barrel for heat accumulation to dry the resin. This effort was not successful as the extruded foam product varied from dry to sporadic slugs of wet material. So it was necessary to dry the resin prior to use by passing 60-70°F desiccant dried air over it and storing it in moisture proof cans as polyethylene bags will not protect the dried resin against moisture for more than 24 hours.

It is recommended that the exposure time during blending be minimized and the feed hopper be covered or that a hopper drier be utilized, especially during lengthy production runs, to ensure feed material of sufficient dryness.

2.6.2 Foam Collapse - Some foam specimens experienced structural collapse during initial extrusion runs. The collapse was due partly to the moisture in the resin, as previously stated, and partly to the temperature of the extruded material. If the temperature in the final extrusion stage is too hot, the extruded material will not have the time to cool sufficiently to provide the cohesive strength necessary to contain individual gas pockets. The gas bubbles can migrate together and form large gas masses within the extrusion which cannot support themselves and the walls eventually collapse.

This problem was corrected by operating the final extrusion chamber at a lower temperature to allow the foam cells to harden more quickly and achieve the necessary strength.

2.6.3 High Density - Although it is necessary to lower the temperature in the final extrusion stage to avoid foam collapse, too low an extrusion temperature is also a critical variable in production of the water soluble foam. If the temperature of the extruded material is too cold, foaming is inhibited because the material is too hard to allow the gas to expand properly and a high density material results. The density of developmental specimens averaged 5.08 lbs per cu. ft. with a range of 3.58 to 10.0 lbs per cu. ft. (80% were less than 6.0 lbs per cu. ft.). Production specimens averaged 5.19 lbs per cu. ft. ranging from 4.61 to 5.70 lbs per cu. ft.

The proper extruded material temperature for the subject formulation, extrusion equipment and conditions of this investigation are shown in Table 2. However, the temperature variable is critical enough to

TABLE 2 — EXTRUSION PRESS DATA AND CONTROL SETTINGS

Extruder Type	Extro (Pilot Plant)	Mini-Extro (Laboratory)
Screw Diameter, Inches		
Stage 1 (Primary)	2-1/2	1-1/4
Stage 2 (Cooling)	2-1/2	1-1/2
Test Sample No.	Development No. 3L	Production No. 13d
Blowing Agent Type (Conc.)	Freon 12 (100%)	Freon 12 (70%)/Freon 11 (30%)
Resin/Batch No.	Klucel J/X19692-44	Klucel J/X19692-55
Nucleating Agent/Conc.	Talc/0.25%	Talc/0.30%
Extruder Capacity, lbs/hr	150	24
Polymer Throughput Rate, lbs/hr	63.4 (480 g/min)	23.8 (180 g/min)
Blowing Agent, lbs/hr	6.01 (45.5 g/min)	1.93 (14.6 g/min)
Blowing Agent, % of Throughput	9.5	8.1
Blowing Agent Injection Pressure, psi	3500	2000
Stage 1		
Feed Screw, RPM/AMP	16/37	126/12.5
Barrel Temp Zone 1, °F	230	270
Barrel Temp Zone 2, °F	250	300
Barrel Temp Zone 3, °F	300	310
Barrel Temp Zone 4, °F	300	N/A
Barrel Temp Zone 5, °F	325	N/A

TABLE 2 (Continued)

Stage 2		
Cooling Screw, RPM/AMP	16/7	31/6.8
Barrel Temp Zone 1, °F	300	245
Barrel Temp Zone 2, °F	300	220
Barrel Temp Zone 3, °F	250	N/A
Barrel Temp Zone 4, °F	240	N/A
Melt Temp, Coupling Zone, °F	325	320
Die Adapter Pressure, psi	500	950
Die Lip Temp, °F	250	255
Die Size, inches	15/64 Round	1/8 Round

21

...qui... ... 't be reestablished by preproduction trial before any
p...uction run.

2.7 Compressive Stiffness Testing

This test was used to screen likely candidate samples extruded
during the material and processing development stage

2.7.1 Test Description - The compressive stiffness of the loose-fill
material was determined during Phase I using an Instron test machine
(Figure 10) in accordance with Federal Specification PPP-C-850D, Amend-
ment 1 and Interim Amendment 2. The height of all specimens tested was
1.000 +0.040 inches after machining flat to assure even contact with the
platens of the test machine. The other dimensions of the specimen were
dependent on the most convenient size which could be obtained from the
samples produced by the extruder. These dimensions varied from approxi-
mately 1/2 to 1-1/4 inches in diameter. All dimensions were taken with
a standard caliper graduated in 0.002 inch increments. The specimen
size was an exception taken to the 4 x 4 x 1 inch requirement of Federal
Specification PPP-C-850D.

After conditioning at room ambient conditions for a minimum of 24
hours, the specimens were weighed on an enclosed beam balance.

Each specimen was placed in the Instron with the machined surfaces
parallel to the platens and aligned along their vertical centerline. The
platens were then brought together to just touch the specimen's upper
surface with an application of approximately 0.025 psi load (Figure 11)
With the test machine adjusted to apply load at a rate of 0.5 inch per
minute per inch of specimen thickness and a recording chart set to
traverse 10 inches per minute, the specimens were compressed through
loads equivalent to 50 psi.

To determine the effect of the more dense outer coating (skin) of
the extruded specimens on compressive stiffness, both cutout cubes
having no skin as well as right cylinder specimens with skin intact
were tested. In addition, groups of four specimens held lightly
together with a rubber band to provide greater total surface area were
tested for the correlation of compressive stiffness values of individual
specimens when specimen diameters were less than one inch.

2.7.2 Test Results - The resulting compression charts taken from
the test machine recorder were used to determine the strain values for
ascertaining compliance with Federal Specification PPP-C-850D, Class 4
compressive stiffness requirements. The strain values required for
stresses of 20, 40 and 50 psi are shown in Table 3, including the
fifteen percent allowable tolerance. The experimental strain values
were obtained by calculating the actual load required on the specimen
face area to attain the 20, 40 and 50 psi stress values using the
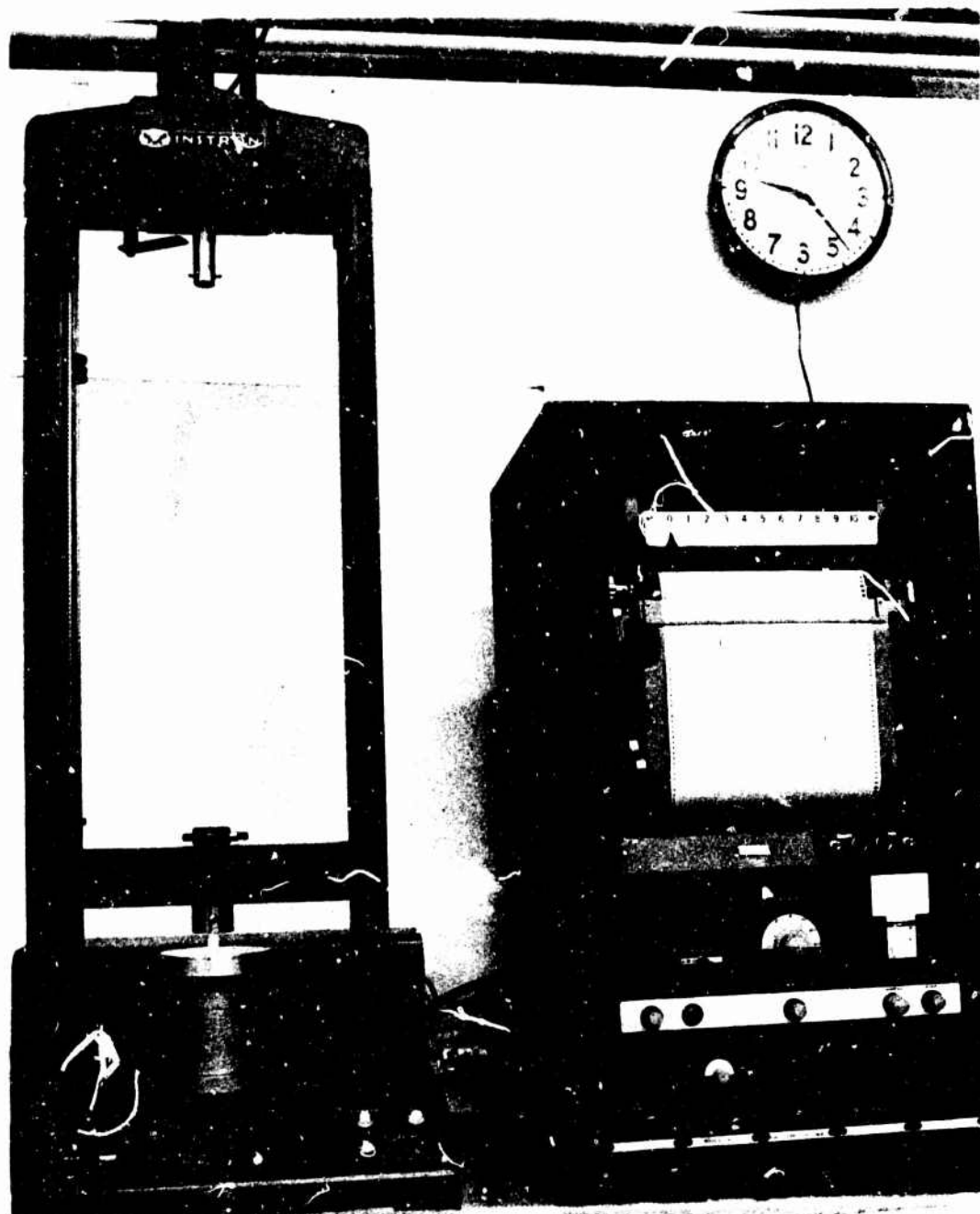following relationship:

FIGURE 10 - INSTRON TEST MACHINE FOR COMPRESSIVE STIFFNESS TESTING

23
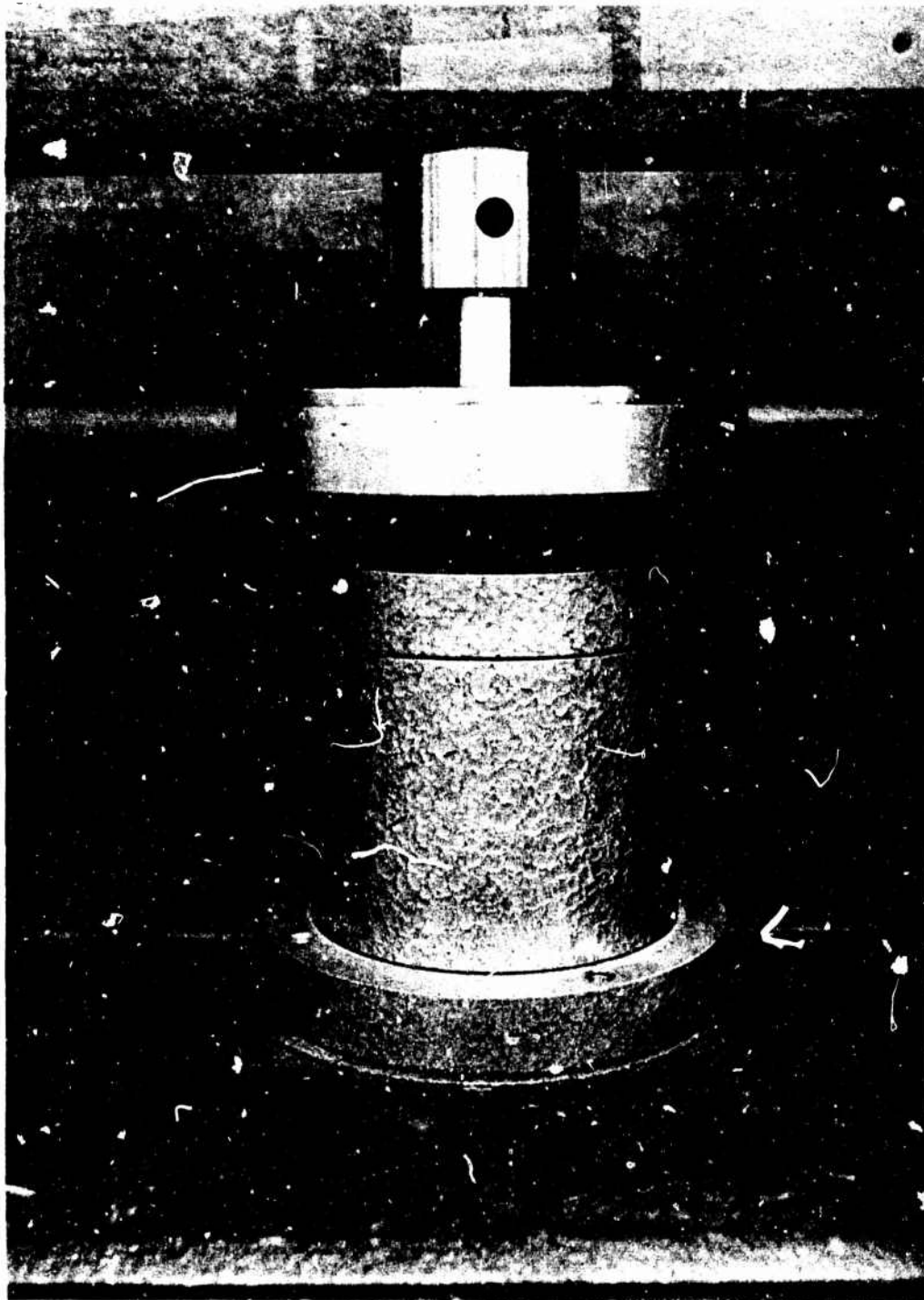
FIGURE 11 - INSTRON TEST MACHINE
PLATENS WITH COMPRESSIVE STIFFNESS SPECIMEN IN POSITION

24

$$L = S \times A$$

where L = Instron load applied, pounds

S = Stress requirement (20, 40 or 50 psi)

A = Specimen surface area in contact with platen, square inches

After the loads were obtained for the three stress values, corresponding deflection values were determined from the compression curve. The strain values were obtained by correcting the deflection values for the specimen thickness, t, as follows:

$$e = \frac{d}{t}$$

where e = Strain value, inches/inch

d = Deflection at load, inches

t = Original specimen thickness, inches.

2.7.3 Discussion - A hydroxypropyl cellulose formulation and process control settings were developed which yielded compressive stiffness values conforming to Federal Specification PPP-C-850D, Class 4 requirements. In most cases, the specimens were found to also meet Class 3 stiffness requirements, which is not unusual due to the 58-85 percent overlap in requirement values, including tolerance (Table 3). However, the tested specimens often were too stiff for the Class 3, 20 psi stress requirement and usually were more centrally positioned within the Class 4 stiffness range for 40 and 50 psi stress applications. For these reasons the loose-fill packaging foam presented for reliability determination and further testing was designated as Class 4 material (Table 4).

The reproducibility of strain values determined for sister specimens was excellent, varying by no more than six percent.

A typical stress/strain curve, as shown in Figure 12, reflects the initial resistance of the loose-fill material to compression. Continued application of pressure resulted in a relaxation or "give" in the material prior to an asymptotic rise in strain. Compression data obtained in this study is not sufficient to explain the portion of the curve in which the strain decreased. This phenomenon was present regardless of the configuration (cube or cylinder) or size of the specimens investigated.

TABLE 3 FEDERAL SPECIFICATION PPP-C-850D COMPRESSIVE STIFFNESS REQUIREMENTS

STRESS (PSI)	STRAIN (IN./IN.)					
	CLASS 3			CLASS 4		
	-15%	0	+15%	-15%	0	+15%
20	.41	.48	.55	.36	.42	.48
40	.53	.62	.71	.54	.64	.74
50	.56	.66	.76	.60	.70	.81

TABLE 4 COMPRESSIVE STIFFNESS VALUES OF FOAMED HYDROXYPROPYL CELLULOSE RESIN (DEVELOPMENT SPECIMENS)

STRESS (PSI)	STRAIN (IN./IN.)		
	DEVELOPMENT SPECIMEN NO.		
	1-5	3L-1	3L-2
20	.41	.43	.43
40	.70	.67	.67
50	.74	.72	.73
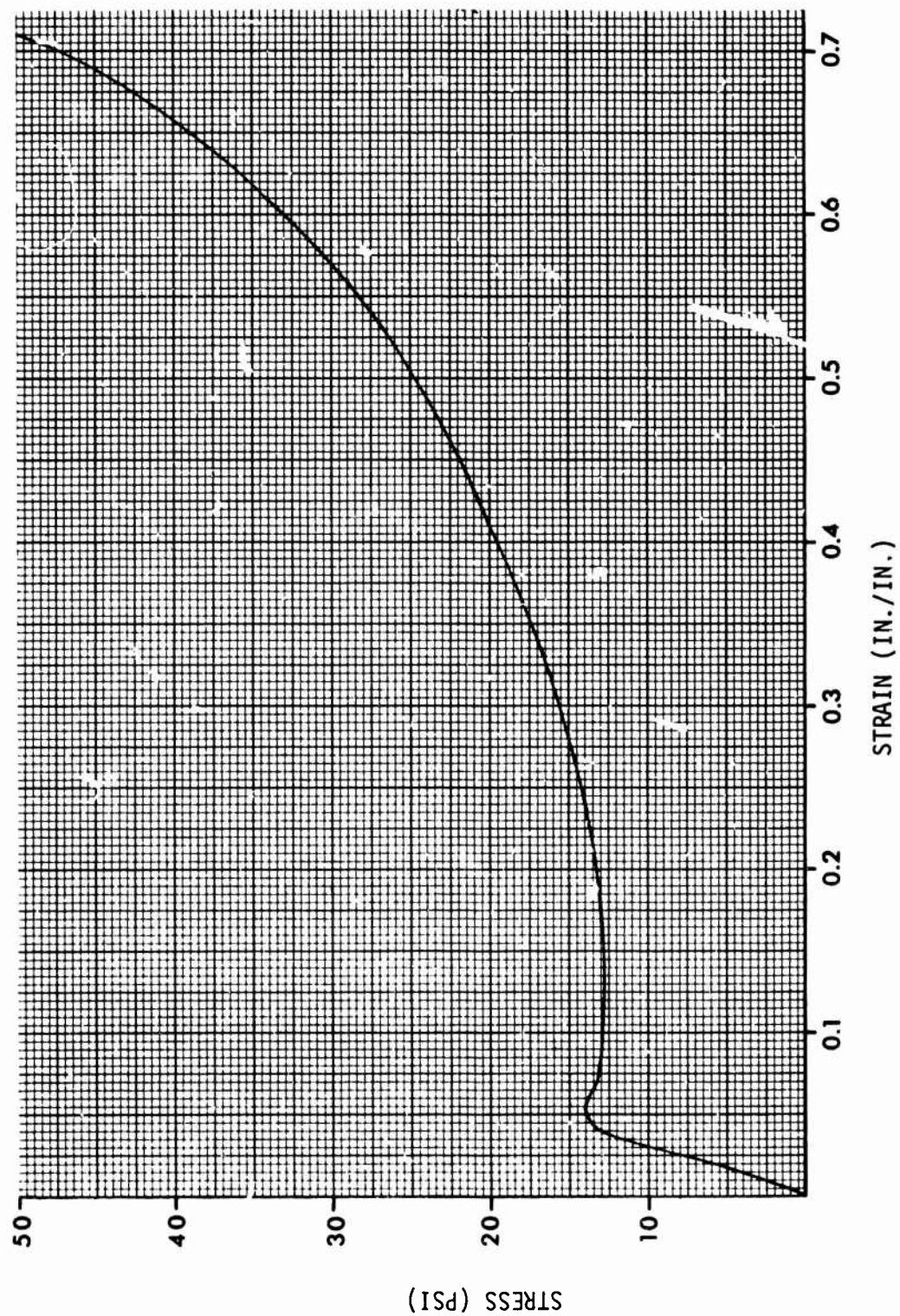
FIGURE 12 - STRESS-STRAIN CURVE (COMPRESSIVE STIFFNESS)

STRESS (PSI)

STRAIN (IN./IN.)

The effect of the more dense outer coating (skin) of the specimens on compressive stiffness values was also determined. Both cutout cubed specimens without skin and cylindrical specimens with skin intact were taken from the same sample section and tested. The results indicated that the presence of the outer skin increased the compressive stiffness value by no more than eight percent, which was not significant enough to cause a specific specimen to change considerably from one stiffness class to another. As a direct result of this test, future compression specimens were tested as right cylinders with skin intact.

The effect of extrusion flow on compressive stiffness was also investigated. Of the skinless cubed specimens tested, some were compressed with the axis of the Instron tester in the machine (extrusion) direction while identical specimens were compressed with the axis in the transverse (right angle to the extrusion) direction. The results indicated a negligible (less than five percent) increase in m sured stiffness in the transverse direction.

Because of the comparatively small (approximately 9/16 inch) diameters of the specimens tested, a group of four specimens held together by a rubber band were tested for compressive stiffness as a group. The excellent correlation between the group specimen and individual specimens from the same extrusion run (reliability run) indicates the validity of using single specimens for stiffness tests (see Table 5).

2.8 Manufacturing Reliability

The ability to reproduce foamed specimens at will, using a pre-established formula and processing parameters to the compressive stiffness requirements of Federal Specification PPP-C-850D, Class 4, was satisfactorily demonstrated. The work was performed using a smaller scale extruder built by the same equipment manufacturer, having the same design features but of lesser throughput capacity than that utilized to obtain the developmental sample data. The fact that previous machine settings found to be satisfactory for earlier work, could be scaled down for the smaller capacity extruder to provide the desired compressive stiffnesses, demonstrated the flexibility of the production process used (see Table 2).

The compressive stiffnesses of the reliability specimens produced, as shown in Table 5, meet the contractural compressive stiffness requirements of Federal Specification PPP-C-850D, Class 4 in its mid-range and very nearly (except for 20 psi stress values) satisfies the PPP-C-850D, Class 3 values as well.

TABLE 5 COMPRESSIVE STIFFNESS VALUES OF FOAMED HYDROXYPROPYL CELLULOSE RESIN

(RELIABILITY SPECIMENS)

STRESS	STRAIN (IN./IN.)						
	RELIABILITY SPECIMEN NO.						
(PSI)	I, II, III, V*	IV	VI	VII	VIII	IX
20	0.40	0.41	0.37	0.40	0.41	0.44
40	0.65	0.66	0.63	0.65	0.65	0.66
50	0.71	0.71	0.70	0.70	0.71	0.71

* Four individual cylinder specimens grouped
  together and compressed simultaneously
  as a unit.

3. PHASE II – PROPERTY DETERMINATION

3.1   PPP-C-1683 Testing

In Phase II, tests were conducted on the loose-fill foam material to determine the physical and mechanical properties when tested as described in Federal Specification PPP-C-1683; Cushioning Material, Expanded Polystyrene, Loose-Fill Bulk. The following procedural test descriptions are in accordance with that specification unless stated otherwise.

3.1.1 Dynamic Test

3.1.1.1 Test Description – A cleated plywood box (Federal Specification PPP-B-601, Style A; Boxes, Wood, Cleated-plywood) was drop tested using the loose-fill packaging material to protect an interior prototype load against shock. The 1/2-inch thick walls of the box were reinforced with 1-inch thick pine cleats at all exterior edges. There were no projections. The interior dimensions of the box were 12 x 12 x 12 inches. A prototype load consisted of a rigid aluminum box with a length, width, and depth of six inches to provide a 36 square inch bearing area on the packaging material. The aluminum box was constructed to allow the installation of ballast weights, which were bolted to the bottom of the box (Figure 13). A triaxial accelerometer, mounted on a rigid post, was located at the geometric center of the prototype load. The ballast weights were designed to provide static stress levels on the packaging material of 0.104 to 0.624 pounds per square inch (psi) in 0.104 psi increments. The inside bottom surface of the plywood box was covered with a three inch layer of the packaging material. The box was briskly rocked from side to side for 30 seconds to provide a uniform layer. Additional material was added, as required, to obtain a three inch depth. The prototype test load was placed on the layer of packaging material and centered. In accordance with the test method (PPP-C-1683), the box was filled to the top with the packaging material and then over-filled with an extra amount of material so that when a 50 pound weight was placed on the cover, a gap of approximately one-eight inch existed between the cover and the box. The cover was then secured with four carriage bolts. An adjustable sling assembly, attached to eye-bolts in the plywood box cover, was used to raise and level the pack assembly. The sling assembly was attached to an electrically operated quick release mechanism which was hung on a chain hoist. The mechanism and box attachment were arranged so that no rotational or sidewise forces were imparted when the box was dropped to the floor.
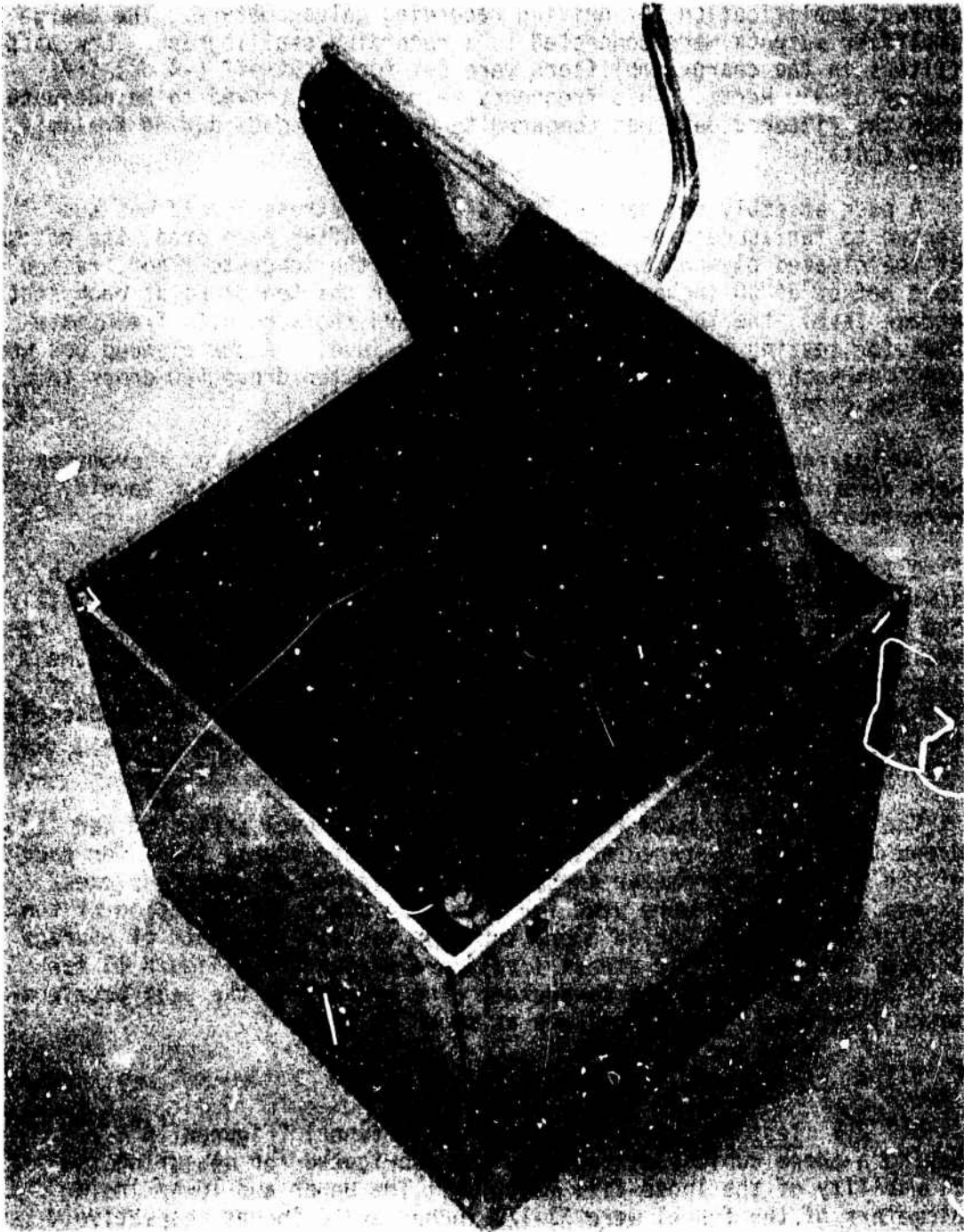
FIGURE 13 - PROTOTYPE LOAD WITH ACCELEROMETER
AND ONE BALLAST WEIGHT INSTALLED

31

The outputs of the triaxial accelerometer were electrically connected to charge amplifiers which provided signal conditioning, filtering and current amplification for driving recording galvanometers. The charge amplifier outputs were connected to a recording oscillograph. Low-pass filters in the charge amplifiers were set for a cut-off (-3 dB) frequency of 330 Hertz. This frequency response was proved to be adequate when the filtered data was compared to unfiltered data during preliminary tests.

A pack assembly for each of the six static stress levels was subjected to ten successive drops (Figure 14). After each drop, the bottom of the cleated plywood box was leveled with the concrete floor, raised to a height of 30 inches and dropped. After the ten drops at each static stress level, the loose-fill was removed and replaced with fresh material for testing at the next static stress level. A new plywood box was used for each series of six bearing loads and ten drops (60 drops total per box used).

During each drop test, the outputs from the triaxial accelerometer were recorded on the oscillograph and the peak acceleration levels were tabulated.

3.1.1.2 Test Results - When the box cover was removed after each series of ten drop tests, it was observed that a space of approximately one inch in depth existed due to settling of the packing material. It was also observed that following each series of drop tests where the static stress level was 0.208 psi or above, noticeable compaction of the packaging material occurred. The packaging material was compacted to the point that it would not fall from the box when the box was inverted.

The acceleration data from the last eight of each series of ten drops tests were averaged and plotted vs. static stress level for each of the three accelerometer axes. Absolute values of the cross-axes (X and Y axes) data were used for averaging since the direction of the acceleration pulses varied among the drop tests. The plots of average acceleration vs. static stress levels are presented in Figure 15 for each series of drop tests conducted. The average of the peak accelerations in the X, Y and Z-axes are as shown in Table VI.

3.1.2 Flowability Test

3.1.2.1 Test Description - An aluminum funnel (Figures 16 & 17) having a three cubic foot capacity was fabricated for measuring the flowability of the loose-fill material. The upper and lower inside diameters of the funnel were 33-1/2 inches and 6 inches respectively and the slope of the wall was 45 degrees. The six inch diameter bottom neck was 12 inches long and was fitted with a nonrestricting, quick opening slide gate. After the funnel was filled with loose-fill
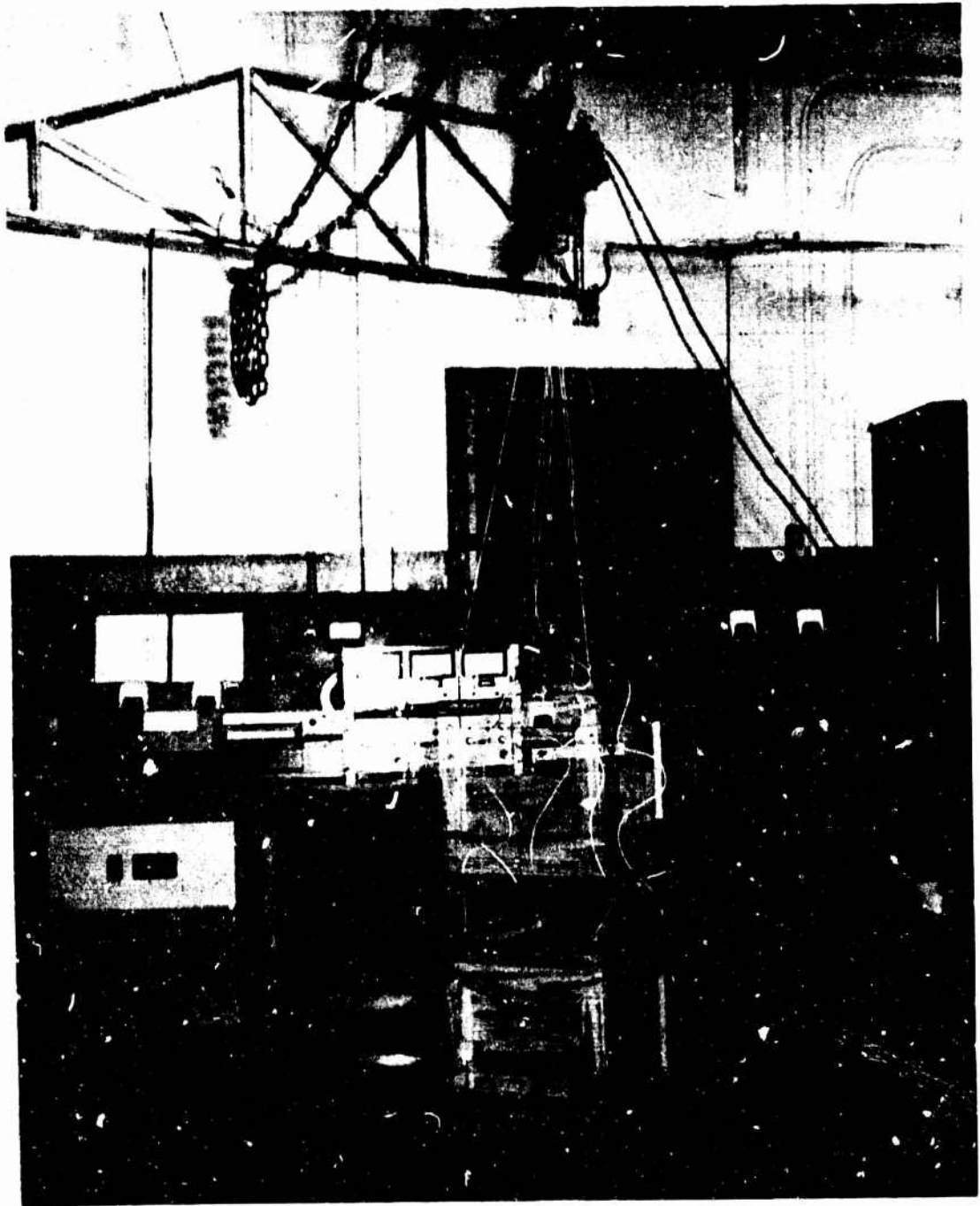
FIGURE 14 - DYNAMIC DROP TEST

33
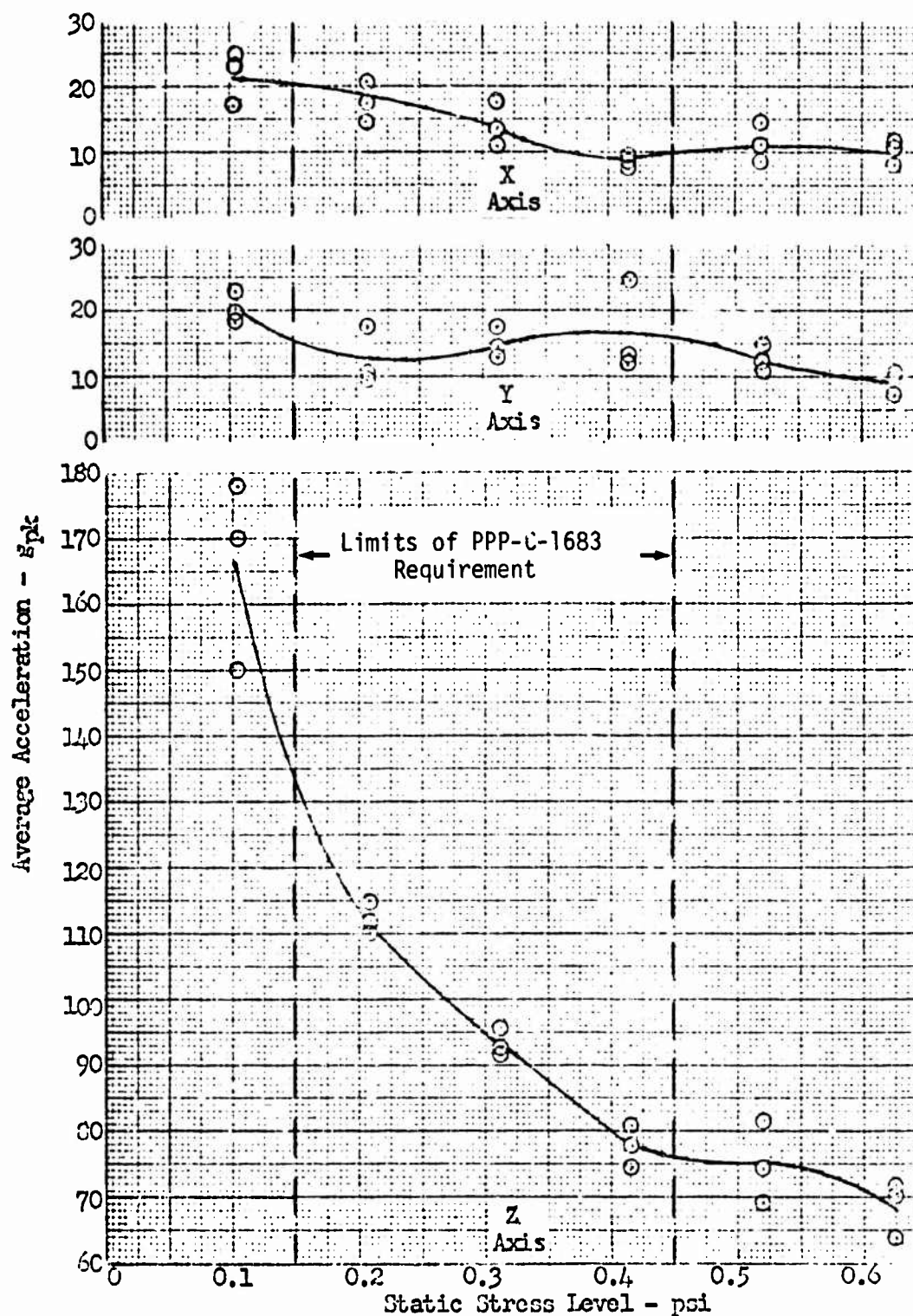
FIGURE 15 - AVERAGE ACCELERATION VS STATIC STRESS LEVEL

34

TABLE 6 PEAK ACCELERATIONS (G'S)*

RUN NO.	AXIS	STATIC STRESS LEVEL (PSI)					
		0.104	0.208	0.312	0.416	0.520	0.624
1	X	23.3	17.3	17.5	7.3	8.4	10.9
	Y	18.1	10.8	14.4	11.7	12.1	7.2
	Z	178	112	95.7	80.6	69.4	63.8
2	X	24.5	14.6	10.6	9.2	11.1	11.2
	Y	19.7	9.5	12.9	13.1	10.5	9.7
	Z	170	110	92.6	74.6	74.5	71.5
3	X	17.0	20.9	13.2	8.8	14.3	8.0
	Y	22.8	17.4	17.4	24.7	14.9	10.6
	Z	150	115	91.6	77.8	81.5	70.2

*Values are averages of drops No. 3 through No. 10.

35
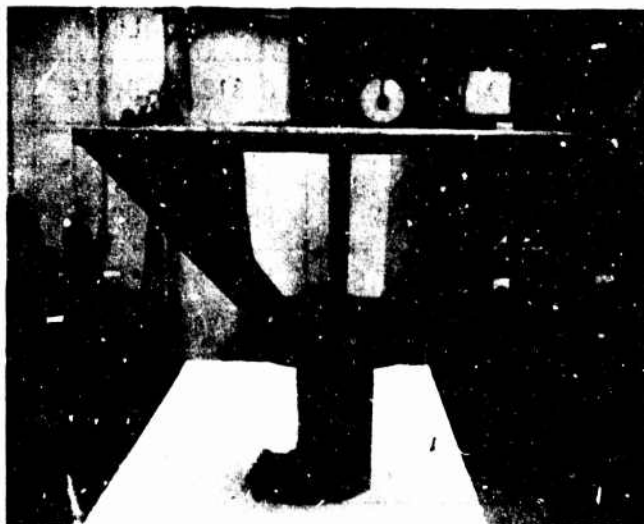
FIGURE 16 - FLOWABILITY TEST FUNNEL

FIGURE 17 - FLOWABILITY TEST FUNNEL
(INTERIOR)

foam material and leveled off (Figure 18), the slide gate was opened
and the m? :rial allowed to flow freely (Figure 19). The time
required ,or the funnel to become empty was determined. Three tests
were performed.

3.1.2.2 Test Results - The elapsed times for the funnel to become
empty are shown Table 7. No loose-fill material was retained in
the funnel.

3.1.3 Vibrational Settling Test

3.1.3.1 Test Description - A test assembly as prepared for the
dynamic test previously described in 3.1.1.1 was used for the vibra-
tional settling test except that the prototype loads tested were
0.156 and 0.468 psi due to equipment limitations. The completed pack
was placed on the head of an electrodynamic force exciter for excita-
tion in the vertical (Z) axis. Fences were bolted to the exciter
head to block the lateral motion of the pack assembly without
restricting its vertical movement. A calibrated piezoelectric
accelerometer was bonded to the exciter head adjacent to the fence
to monitor the applied excitation (Figure 20). The average vertical
distance from the top face of the four corners of the prototype load to
the top of the box was determined and recorded.

Sinusoidal vibratory excitation was applied to each pack assembly
for a period of 30 minutes. An excitation level of 1.1 gpk (0.93 inch,
double-amplitude) was applied at 4.8 Hz. Six 30-minute tests were
conducted; three tests with pack assemblies having the prototype load
ballasted to provide a static stress level of 0.156 psi and three tests
with a 0.468 psi static stress level. The pack assemblies were removed
from the exciter after each test and the packaging material above the
test load was carefully removed. The vertical distance from each of the
four upper corners of the prototype load to the top of the box was
remeasured and recorded.

3.1.3.2 Test Results - The average of the four corner measurements
was used to calculate the vertical displacement (vibrational settling)
as a percentage of the initial distance measured. A summary of the
vibration settling tests is tabulated in Table 8.

It was noted that voids due to packaging material settling and
compaction similar to that observed during free fall drop testing
occurred during the vibrational settling tests.
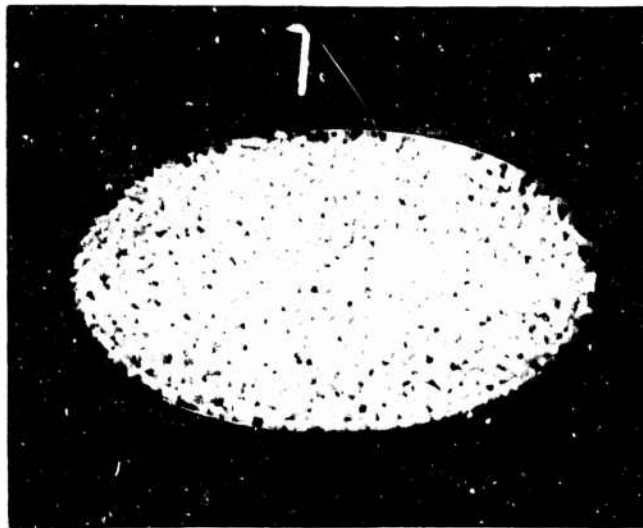
FIGURE 18 - FUNNEL LOADED WITH FOAM
PRIOR TO FLOWABILITY TEST
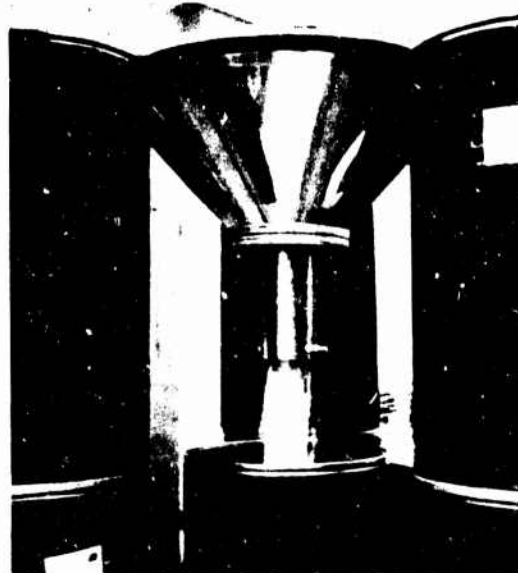
FIGURE 19 - GRAVITY FLOW OF LOOSE-FILL
MATERIAL FROM FLOWABILITY
TEST FUNNEL
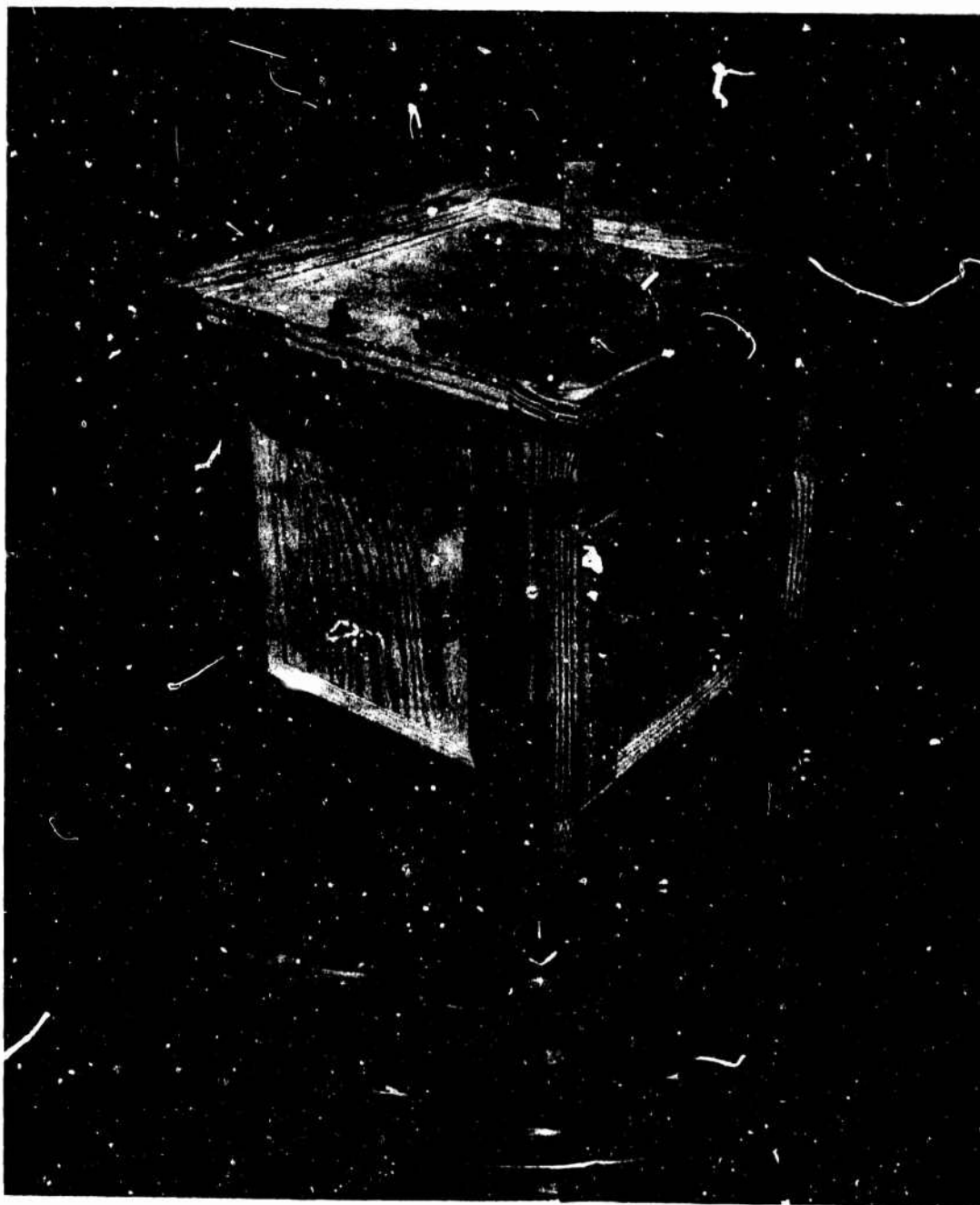
FIGURE 20 - VIBRATIONAL SETTLING TEST

TABLE 7 FLOWABILITY

RUN NO.	ELAPSED FLOW TIME (SEC/3 CU.FT.)	FLOWABILITY CU.FT./MIN
1	13.5	13.3
2	13.4	13.4
3	13.3	13.5
AVG.	13.4	13.4

TABLE 8 VIBRATIONAL SETTLING

TEST NO.	TEST TIME (Min)	EXCITATION FREQUENCY (Hz)	EXCITATION LEVEL		STATIC STRESS (psi)	VIBRATIONAL SETTLING (%)
			(in_{pk-pk})	(g_{pk})		
1	30	4.8	0.93	1.10	0.156	8.0
2	30	4.8	0.93	1.10	0.156	8.0
3	30	4.8	0.93	1.10	0.156	5.0
4	30	4.8	0.93	1.10	0.468	14.0
5	30	4.8	0.93	1.10	0.468	16.0
6	30	4.8	0.93	1.10	0.468	13.7

The vibrational settling tests were conducted per Federal Specification PPP-C-1683 with the following exceptions:

(1) The static stress levels employed (0.156 and 0.468 psi) were four percent above those specified due to equipment limitations.

(2) The excitation frequency of 4.8 Hz employed versus the 4.5 Hz specified for the vibrational settling tests caused the application of an additional 540 cycles of excitation during each 30 minute test period. This amounted to a 6.7 percent increase in the number of test cycles applied.

3.1.4 Loaded Bulk Density Test

3.1.4.1 Test Description - A cleated, plywood box (Figure 21) as described for the dynamic test was used to contain the loose-fill material for the determination of loaded bulk density. After filling the box with the loose-fill material, it was leveled and a 11-7/8 x 11-7/8 x 3/4 inch plywood leveling board placed on top of it. The leveling board was then loaded with 50 pounds of weight at the center (Figure 22). The corner depths from the top of the box to the underside of the leveling board was measured and the average distance determined. The net weight of the loose-fill was measured and the bulk density calculated to the nearest 0.05 pounds per cubic foot. Three runs were made, each using fresh loose-fill material.

3.1.4.2 Test Results - The bulk densities (Table 9) were determined from the following equation:

$$\text{Bulk Density (lbs/cu ft)} = \frac{0.0265 \ W}{11.25 \ + D}$$

where: W = net weight (gms) of loose-fill

D = average corner depth from top of box to underside of leveling board (inches)

3.1.5 Compressive Creep Test

3.1.5.1 Test Description - Compressive creep characteristics of the loose-fill material was determined by maintaining a constant compressive load and measuring the deformation periodically. Two modified Kerstner boxes (Figure 23) each consisting of an inner and outer box of 3/4 inch thick plywood was fabricated for this test.

The inner box, with exterior dimensions of 6-3/8 x 6-3/8 inches by 8 inches deep, served as a movable, guided platen into which the necessary weights were placed to achieve 0.4, 0.8 and 1.2 psi (nominal) loadings. The outer box, serving as a base plate for the testing

FIGURE 21 - CLEATED BOX, LEVELING BOARD
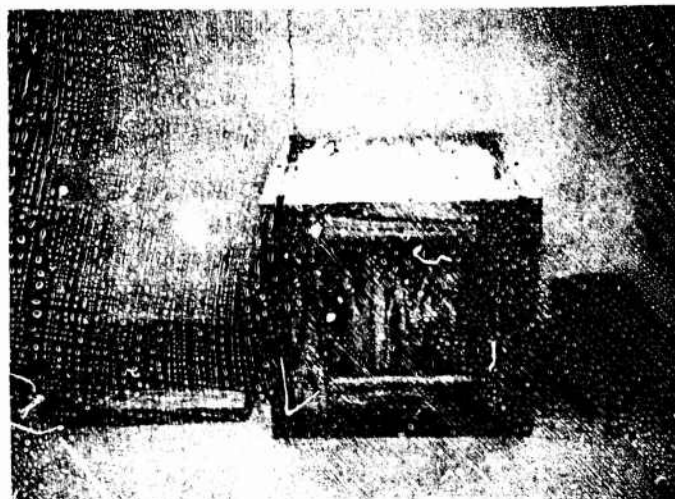AND 50 POUND WEIGHT FOR
LOADED BULK DENSITY TEST

FIGURE 22 - LOOSE-FILL UNDERGOINC
LOADED BULK DENSITY TEST

TABLE 9   LOADED BULK DENSITY

RUN NO.	BULK DENSITY (LBS/CU FT)
1	2.80
2	2.80
3	2.85
AVG.	2.80

device, had inside dimensions of 6-1/2 x 6-1/2 inches by 9-1/4 inches deep. As required by Federal Specification PPP-C-1683, the wood from the back panels were replaced with translucent plastic. The coefficient of friction between the inner and outer boxes was measured and found to be 0.3. An exception was taken to the loose-fill dimensional requirement of PPP-C-1683 for the specimen size. The production material produced for testing was right cylinders approximately one inch high by 9/16 inch diameter rather than the specified "at least 2 x 2 inches along the length and width and one inch in thickness." This change was dictated by the size of the foam extruder available for our use.

The loose-fill material was placed evenly in the outer box to a depth of about 3-1/2 inches and the unloaded inner box gently placed upon it. One of the required loads was then placed into the inner box so as to avoid wedging within the outer box (Figure 24). The average distance between loading surfaces was then measured and recorded as T_i, the initial thickness under load. Subsequent measurements were made after 6 minutes, 1 hour, 24 hours, 96 hours (4 days) and 168 hours (7 days) of load duration. Three tests for each of the three loadings were made.

3.1.5.1 <u>Test Results</u> - The static stress (Table 10) for each load and the compressive creep values (Table XI) were calculated as shown below. The amount of creep observed was less than four percent and is plotted in Figure 25.

$$\text{Static Stress, psi} = \frac{W}{A}$$

where: W = Weight of load applied to inner box in pounds

A = Area of inner box bearing surface in square inches

$$\text{Compressive Creep, percent} = \frac{T - Tn}{T} \times 100$$

where: T = Thickness of original material placed in box in inches

Tn = Thickness of material under load at any particular time interval, n, in inches

3.1.6 <u>Compressive Set Test</u>

3.1.6.1 <u>Test Description</u> - The compressive set characteristics of the loose-fill material was determined by measuring the recovery response of the material upon removal of the loads applied for the Compressive Creep Test after the 168 hour (7 day) period. The thickness of the material after three recovery time intervals, 30 seconds, 30 minutes and 24 hours, were measured.

FIGURE 23 - INTERIOR AND EXTERIOR
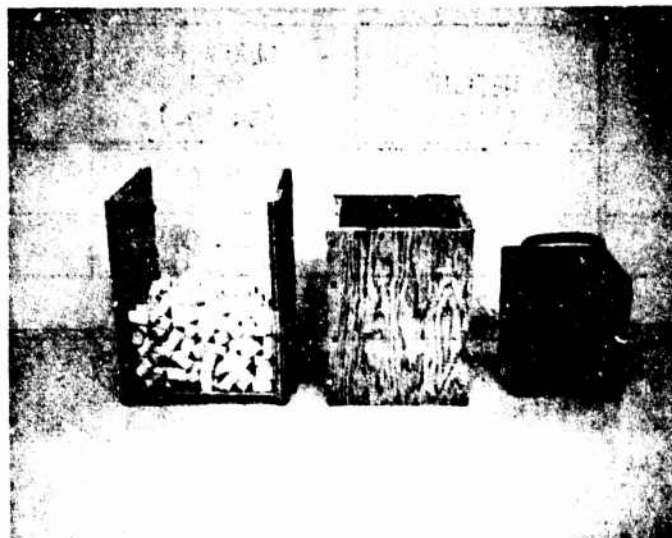MODIFIED KERSTNER BOX
WITH LOOSE-FILL AND WEIGHT
FOR COMPRESSIVE CREEP/SET TEST

FIGURE 24 - LOOSE-FILL MATERIAL
UNDERGOING COMPRESSIVE
CREEP/SET TEST

TABLE 10 TEST LOAD DETERMINATION

PPP-C-1683 REQUIREMENT (psi)	WEIGHT (lbs)	SURFACE AREA (sq in.)	CALCULATED STATIC STRESS (psi)
0.4	18.0	40.64	0.44
0.8	34.0	40.64	0.84
1.2	52 0	40.64	1.28

TABLE 11 COMPRESSIVE CREEP

ACTUAL STATIC STRESS, LOAD (psi)	TIME INTERVAL	ORIGINAL THICKNESS, T (INCHES)	LOADED THICKNESS, Tn (INCHES)	COMPRESSIVE CREEP (%)
0.44	1 min.		3.30	0.00
	6 min.		3.30	0.00
	1 hr.	3.30	3.29	0.30
	24 hrs.		3.27	0.91
	96 hrs.		3.25	1.52
	168 hrs.		3.25	1.52
	1 min.		3.13	0.00
	6 min.		3.13	0.00
	1 hr.	3.13	3.11	0.64
	24 hrs.		3.10	0.96
	96 hrs.		3.09	1.28
	168 hrs.		3.09	1.28
	1 min.		3.25	0.00
	6 min.		3.25	0.00
	1 hr.	3.25	3.23	0.62
	24 hrs.		3.20	1.54
	96 nrs.		3.20	1.54
	168 hrs.		3.20	1.54
0.84	1 min.		3.19	0.00
	6 min.		3.19	0.00
	1 hr.	3.19	3.15	1.25
	24 hrs.		3.13	1.88
	96 hrs.		3.13	1.88
	168 hrs.		3.13	1.88
	1 min.		3.38	0.00
	6 min.		3.38	0.00
	1 hr.	3.38	3.34	1.18
	24 hrs.		3.31	2.07
	96 hrs.		3.31	2.07
	168 hrs.		3.31	2.07
	1 min.		3.23	0.00
	6 min.		3.23	0.00
	1 hr.	3.23	3.19	1.24
	24 hrs.		3.16	2.17
	96 hrs.		3.16	2.17
	168 hrs.		3.16	2.17

TABLE II COMPRESSIVE CREEP (CONT'D)

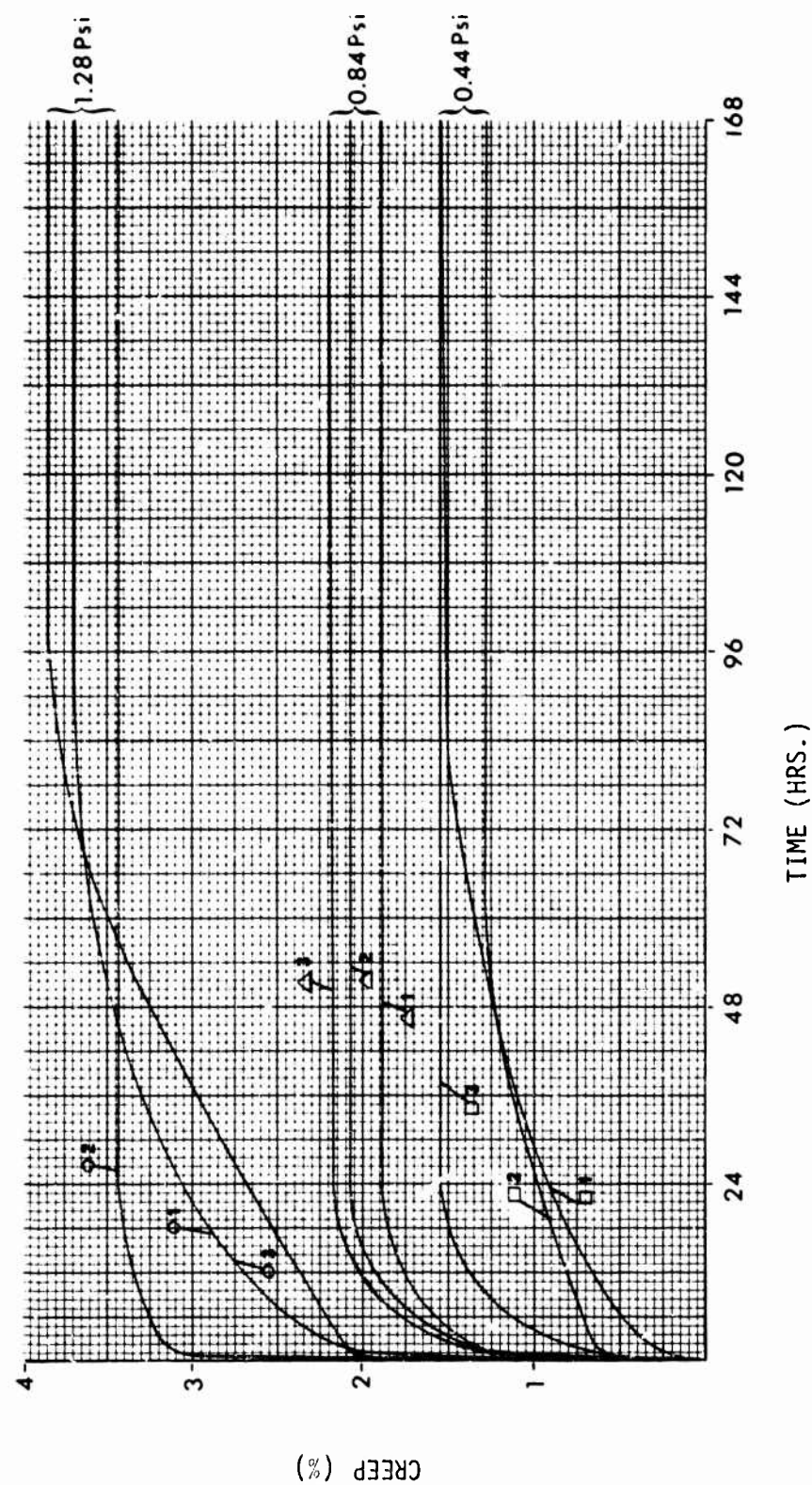| ACTUAL STATIC STRESS, LOAD (psi)	TIME INTERVAL	ORIGINAL THICKNESS, T (INCHES)	LOADED THICKNESS, Tn (INCHES)	COMPRESSIVE CREEP (%)
1.28	1 min.	3.25	3.25	0.00
	6 min.		3.25	0.00
	1 hr.		3.19	1.85
	24 hrs.		3.15	3.08
	96 hrs.		3.13	3.69
	168 hrs.		3.13	3.69
	1 min.	3.50	3.50	0.00
	6 min.		3.50	0.00
	1 hr.		3.39	3.14
	24 hrs.		3.38	3.43
	96 hrs.		3.38	3.43
	168 hrs.		3.38	3.43
	1 min.	3.38	3.38	0.00
	6 min.		3.38	0.00
	1 hr.		3.31	2.07
	24 hrs.		3.29	2.66
	96 hrs.		3.25	3.84
	168 hrs.		3.25	3.84

FIGURE 25 - COMPRESSIVE CREEP CURVE

49

3.1.6.2 Test Results - Upon removal of the load applied to the loose-fill material on completion of the 168 hour Compressive Creep Test, no measurable thickness change (recovery) of the compressed material took place during the 24 hour test period. Consequently, the percentage compressive set after the 30 second, 30 minute and 24 hour test intervals was the same value as that obtained at the end of the 168 hour Compressive Creep Test as shown in Table 12.

3.1.7 Flammability Test

3.1.7.1 Test Description - A tared basket, 8 inches long by 6 inches wide by 8 inches tall was fabricated of 1/4 inch stainless steel wire mesh for testing the flammability of the loose-fill material. After filling the basket with loose-fill material, a one foot length of MIL-F-18405; Fuse, Firecracker, 30-second was threaded through the center of the six inch basket width and allowed to protrude from each side (Figure 26). Additional loose-fill was then added to the basket until level with the top. The filled basket was then weighed and the net weight of the contents, less one gram for the fuse, was determined. The filled basket was then placed in a draft free environment and the fuse ignited. After the fuse had been consumed, the basket and contents were reweighed. Seven tests were performed.

3.1.7.2 Test Results - The flammability of the loose-fill material, based on the percentage of net loose-fill remaining after the test, was calculated as follows:

Percentage material remaining = $\dfrac{\text{Final net weight, gms}}{\text{Original net weight, gms}}$ x 100

The results are shown in Table 13.

3.1.8 Electrostatic Adhesion Test (Figures 27 thru 31)

3.1.8.1 Test Description - The loose-fill material was tested for electrostatic adhesion properties by filling a five gallon, 11 inch diameter fiberboard drum with telescoping cover, one-third full of material. While lying on its side, the drum was rolled back and forth across a ten foot span of floor for three minutes at a rate of approximately 60 revolutions per minute. Within 30 seconds after the final revolution, the drum was opened and the loose-fill allowed to empty out by gravity (without shaking).

In addition to the preceding PPP-C-1683 test procedure, the same drum, again filled to one-third capacity, was placed on a power driven roller and rotated at the 60 rpm rate for the three minute test.

Test Results - Three tests were performed for each of the above test procedures. In each test no loose-fill material was retained in the emptied drum.

TABLE 12 COMPRESSIVE SET

ACTUAL STATIC STRESS, LOAD (psi)	DURATION OF LOAD (hrs)	THICKNESS CHANGE (inches)	RECOVERY TIME	COMPRESSIVE SET (%)	
0.44	168	0.00	30 sec, 30 min & 24 hrs.	1) 2) 3)	1.52 1.28 1.54
0.84	168	0.00	30 sec, 30 min. & 24 hrs	1) 2) 3)	1.88 2.07 2.17
1.28	168	0.00	30 sec, 30 min. & 24 hrs.	1) 2) 3)	3.69 3.43 3.84

TABLE 13 FLAMMABILITY

TEST NO.	NET WEIGHT OF LOOSE-FILL (GRAMS)		MATERIAL REMAINING (%)
	INITIAL	FINAL	
1	281	280	99.6
2	285	284	99.7
3	283	282	99.7
4	285	284	99.7
5	283	283	100.0
6	283	281	99.3
7	285	284	99.7
AVERAGE	---	---	99.7
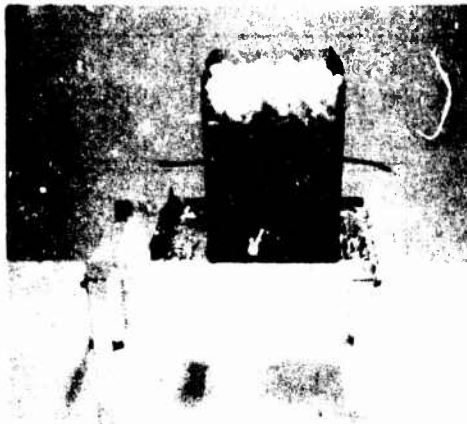
WIRE BASKET AND
LOOSE-FILL MATERIAL
WITH FUSE PROTRUDING

STEP 1
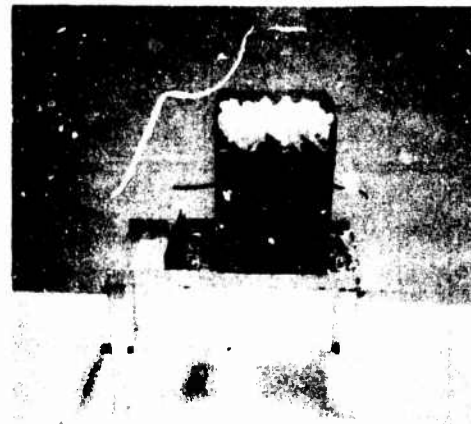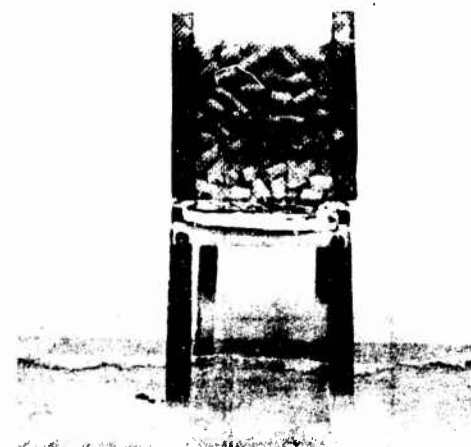
IGNITION OF FUSE

STEP 2

FUSE BURNING THROUGH
LOOSE-FILL MATERIAL

STEP 3

COMPLETED TEST
WITH BURNED
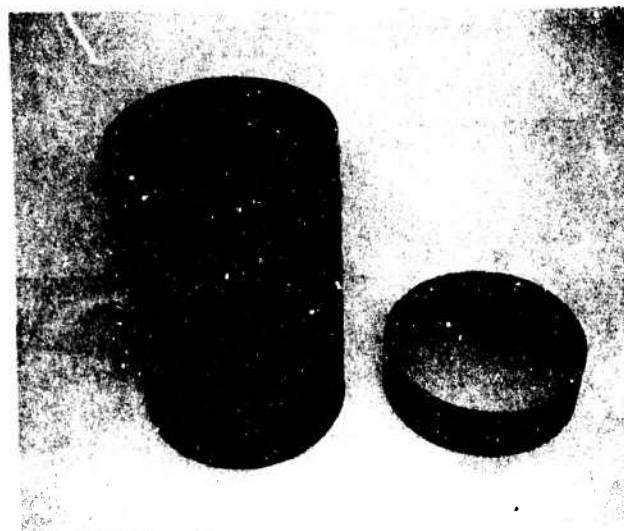CENTRAL AREA

STEP 4

FIGURE 26 - FLAMMABILITY TEST

FIGURE 27 - FIVE GALLON, ALL FIBER
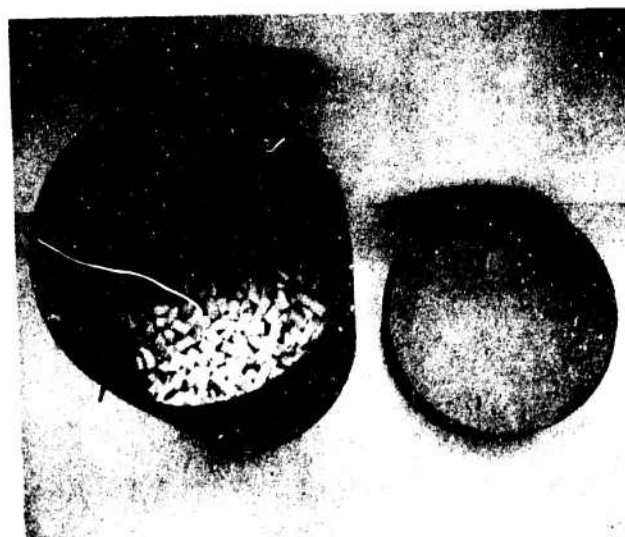DRUM WITH TELESCOPING LID
FOR ELECTROSTATIC ADHESION TEST

FIGURE 28 - ELECTROSTATIC ADHESION TEST
DRUM 1/3 FULL OF TEST MATERIAL

FIGURE 29 - ELECTROSTATIC ADHESION TEST
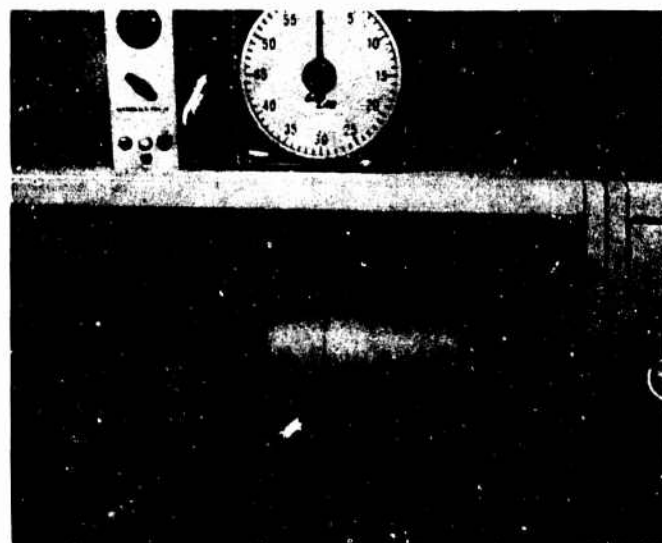DRUM ROLLED PER PPP-C-1683
TEST METHOD

FIGURE 30 - ELECTROSTATIC ADHESION TEST
DRUM ROLLED PER MCDONNELL DOUGLAS
TEST METHOD

FIGURE 31 - EMPTYING OF LOOSE-FILL
MATERIAL AFTER ELECTROSTATIC
ADHESION TEST

3.1.9 **Miscellaneous Examinations** - The loose-fill material was examined for color, configuration, dimensions, tolerances and workmanship. The results are as shown in Table 14..

TABLE 14 MISCELLANEOUS EXAMINATIONS

PROPERTY	RESULT
Color	White
Configuration	Homogeneous right cylinders
Dimensions	Approximately 9/16 inch diameter by 1 inch long
Dimensional Tolerance	+6.6% on diameter (21 random production samples)
Workmanship	Clean, dustless, no visible foreign matter, imperfections or fragmented particles

3.2 Supplemental Testing

The effects of 24 hour and 31 day humidity exposures on product properties were determined and the dissolution rates in salt and fresh water were established.

3.2.1 31 Day Humidity Test

3.2.1.1 **Test Description** - Twenty one specimens of loose-fill foam measuring approximately 9/16 inch diameter by one inch long, each weighing about 0.3 grams (Figure 32), were put into individual aluminum dishes and placed in a humidity cabinet (Figure 33) for 31 days. The environment in the cabinet was controlled at 100 +2°F and 95 +3% relative humidity. The wet and dry bulb temperatures were continuously monitored by a recorder.

3.2.1.2 **Test Results** - At the termination of the 31 day test period, the specimens were removed from the cabinet and reweighed. Each specimen increased in weight an average of 16 percent (Table 15). The appearance of the specimens had changed from an opaque white to a clear, translucent color. The cylindrical cross-section had collapsed and became ellipsoidal and the material was soggy but had not flowed freely (Figure 34).

56

FIGURE 32 - LOOSE-FILL SPECIMENS PRIOR TO 31 DAY HUMIDITY TEST
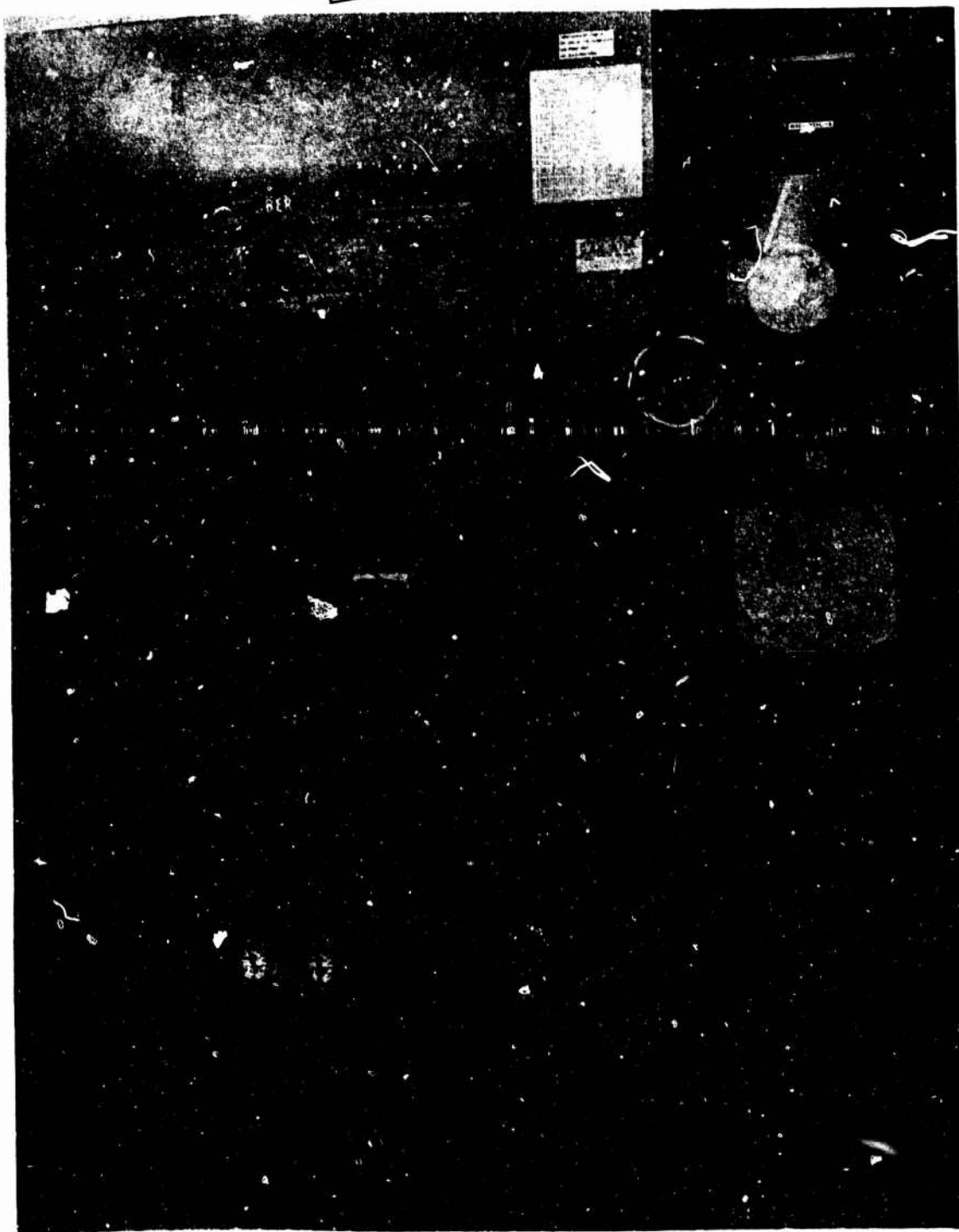
FIGURE 33 - HUMIDITY TEST CABINET

58
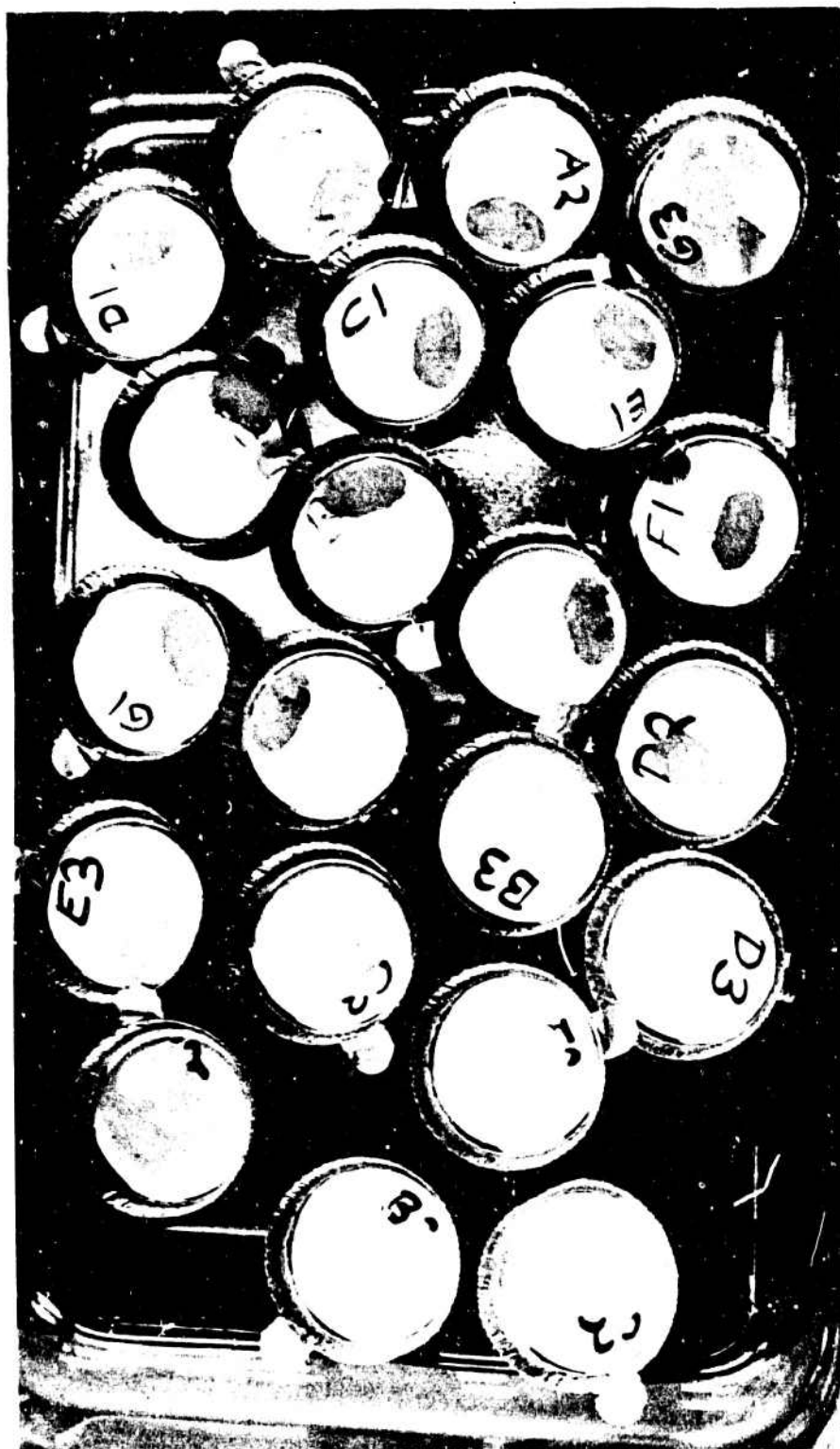
FIGURE 34 – LOOSE-FILL SPECIMENS AFTER 31 DAY HUMIDITY TEST

TABLE 15 31 DAY HUMIDITY EXPOSURE

RUN NO.	SPECIMEN WEIGHT (GMS)*			WEIGHT CHANGE* (%)
	ORIGINAL	FINAL	CHANGE	
1	0.3322	0.3856	0.0534	+16.07
2	0.3387	0.3928	0.0541	+15.97
3	0.3336	0.3869	0.0533	+15.97
AVG.	0.3348	0.3884	0.0536	+16.01

*Average of seven specimens

3.2.2 Moisture Absorption

3.2.2.1 Test Description - Twenty-four hour temperature/humidity exposures were run on foam specimens at 73°F and 50% RH, 73°F and 95% RH, 165°F and 50% RH and 165°F and 95% RH conditions. The weight for each specimen was determined prior to its placement in the environmental chamber.

3.2.2.2 Test Results - As shown in Table 16, specimens exposed to an environment of 73°F and 50% RH for 24 hours experienced an increase in weight of 2-3 percent with slight dimensional changes. Exposure to 73°F and 95% RH resulted in a 7-8 percent increase in weight with more significant dimensional changes in length but not diameter. After exposure to 165°F and 50% RH for 24 hours, specimens had a loss in weight of 3-4 percent, a slight increase in diameter and approximately a 15-20 percent increase in length. On the other hand, after exposure to 165°F and 95% RH, the specimens experienced a 6-7 percent weight gain, had become translucent and had flattened out to an ellipsoidal shape.

3.2.3 Solubility/Dissolution Rate Testing

3.2.3.1 Test Description - Solubility tests were conducted to determine the relative rate of dissolution of a given mass of foam material in given quantities of fresh and prepared sea water. Tests were run on seven sets of specimens in each test group. Specimens in one group were separately placed in glass jars containing 100 ml of fresh (tap) water and were allowed to float freely on the surface. Another group of specimens was individually placed in 100 ml of fresh water and maintained in a submerged state by enclosure in an outer wrap of stainless steel wire mesh. These same procedures were used for two other groups of seven specimens which were floated and submerged in glass jars filled with laboratory prepared sea water containing 2.7 percent sodium chloride (Reference 7).

The pH and temperature of the water solutions in which each specimen was placed were taken before and after dissolution. All specimens were weighed before testing. Lids were used to cover the containers of all floating specimens in order to prevent the specimens from sticking to the wall of the container due to evaporation of the water during the 24 hour test period.

3.2.3.2 Test Results - Twenty-eight hours were required to completely dissolve all floating specimens in fresh water and 32 hours for the specimens exposed to the sea water. Submerged specimens dissolved more rapidly due to the greater contact area which allowed continual dissolution of the gel type outer coating formed during the dissolution process (Reference 8). All of the specimens submerged in fresh water were in solution after ten hours. Twelve hours were required to completely dissolve submerged sea water specimens.

7. Reinhold Publishing Corporation, New York, Condensed Chemical Dictionary, (7th Edition), pg. 837.

8. J. B. Titus, "Environmentally Degradable Plastics," Materials Engineering, April 1974, pg. 20.

TABLE 16   MOISTURE ABSORPTION - SHORT TERM

ENVIRONMENT CONDITIONS TEMP/HUMIDITY	SPECIMEN BEFORE EXPOSURE			SPECIMEN AFTER EXPOSURE			MOISTURE ABSORPTION (PERCENT CHANGE)		
	WEIGHT (GRAMS)	DIAMETER (INCHES)	LENGTH (INCHES)	WEIGHT (GRAMS)	DIAMETER (INCHES)	LENGTH (INCHES)	WEIGHT	DIAMETER	LENGTH
73°F, 50% RH	0.3244	0.578	0.968	0.3335	0.588	0.989	2.81	1.73	2.17
	0.3274	0.574	0.986	0.3366	0.590	0.984	2.81	2.79	-2.03
	0.3295	0.572	0.988	0.3385	0.586	1.005	2.73	2.45	1.72
73°F, 95% RH	0.3237	0.579	0.990	0.3475	0.577	1.070	7.35	-0.35	8.08
	0.3283	0.569	1.001	0.3519	0.568	1.093	7.19	-0.18	9.19
	0.3215	0.578	0.982	0.3441	0.576	1.060	7.03	-0.35	7.94
165°F, 50% RH	0.3193	0.576	0.977	0.3089	0.595	1.153	-3.26	3.30	18.01
	0.3226	0.580	0.978	0.3123	0.607	1.157	-3.19	4.66	18.30
	0.3152	0.572	0.981	0.3048	0.599	1.127	-3.30	4.72	14.88
165°F, 95% RH	0.3223	0.570	0.993	0.3430	*	*	6.42	*	*
	0.3209	0.586	0.982	0.3414	*	*	6.39	*	*
	0.3275	0.578	0.989	0.3476	*	*	6.14	*	*

*Unmeasurable because of specimen collapse.

Changes in test fluid temperatures during the testing were negligible. The average temperature decreased from 62.5°F to 61°F. Changes in the pH of the fluid media are shown in Table 17. In each case, the pH of the solutions which had contained the submerged specimens were lower than those for floating specimens.

TABLE 17   DISSOLUTION RATE

| FLUID MEDIA | SOLUTION PH | | | |
| | BEFORE DISSOLUTION | | AFTER DISSOLUTION | |
	FLOATING	SUBMERGED	FLOATING	SUBMERGED
Fresh Water	9.85	9.85	8.8	8.4
Sea Water	9.30	9.30	8.1	7.5

4. SUMMARY AND CONCLUSIONS

The results of the feasibility study for Phase I show that hydroxy-propyl cellulose resin can be satisfactorily formulated and processed into a loose-fill foam material using a conventional extrusion process. A candidate packaging foam was produced which met the compressive stiffness requirements of Federal Specification PPP-C-850D for Class 4 (Extra Firm) material. During development, foamed products in all other specification classes were also produced using small variations in formulation and machine settings to effect the changes.

The density of the loose-fill produced was relatively high for a plastic foam material used in packaging applications. In addition to higher costs due to greater resin content, the effect of high density was reflected in loaded bulk density and certain peak acceleration properties which did not comply to the requirements of Federal Speci-fication PPP-C-1683.

The 9/16 inch diameter by one inch long loose-fill material was extremely flowable, had excellent vibrational settling characteristics and exhibited no residual electrostatic charge.

Although the material possessed strong resistance to compressive creep for loadings up to 1.2 psi, when the loads were removed no measurable recovery took place within the 24 hour test period. Still, the compressive set values were well within the Federal Specification PPP-C-1683 requirements due to the small amount of compression that occured during the creep test.

Flammability tests showed that the foamed hydroxypropyl cellulose did not support combustion, experiencing only a minute amount of weight loss due to consumed material. During the burning process, the resin in contact with the fuse was degraded from a normal white, fiberous condition to a fused brown mass.

The water soluble characteristic of the resin was not adversely affected by either the formulation or the extrusion process. Complete dissolution in both fresh water and prepared sea water was demonstrated in both the floating and submerged states. Complete solubility in sea water took a slightly longer time than in fresh water exposures.

Long term conditions of 100°F and 95 percent RH caused the specimen surfaces to become tacky and to enlarge an appreciable amount as moisture was absorbed. Specimens that were exposed to shorter term moisture absorption tests behaved in a similiar manner provided the temperature was not elevated. At a temperature of 165°F and 50 percent

relative humidity, the specimens experienced a weight loss as the surrounding heat drove out moisture. However, at 165°F and 95 percent relative humidity, the specimens again absorbed moisture in spite of the hot environment due to the greater amount of moisture present at the higher humidity.

Table 18 compares the physical and mechanical property requirements of Federal Specification PPP-C-1683 with the average values obtained from the loose-fill production material when tested in accordance with the subject specification.

TABLE 18 FEDERAL SPECIFICATION

PPP-C-1683 TEST SUMMARY

NAME OF TEST	PPP-C-1683 REQUIREMENT	TEST RESULT
1. Dynamic	Type I: 60g maximum peak accelerations within 0.15-0.45 psi static stress range.	X-axis = 20.5 g's, max. Y-axis = 16.5 g's, max. Z-axis = 133 g's, max.
	Type II: 90g maximum peak accelerations within 0.15-0.45 psi static stress range.	
2. Flowability	Class 1: 6.0 cu ft per minute or greater. Class 2: Less than 6.0 cu ft per minute	13.4 cu ft per minute (Class 1)
3. Color	Type I: White only Type II: Other than white	White (Type I)
4. Configuration	Supplier's option but homogeneous	Homogeneous right cylinders

TABLE 18   FEDERAL SPECIFICATION
PPP-C-1683 TEST SUMMARY (Continued)

NAME OF TEST	PPP-C-1683 REQUIREMENT	TEST RESULT
5. Dimensions	No overall outside dimension greater than 2 inches (if other than strand configuration)	Approximately 9/16 inches diameter by 1 inch long
6. Tolerance (dimensional)	+20% (if other than strand configuration)	+6.6% on diameter of 21 random production samples
7. Vibrational Settling	30% maximum displacement	7% avg. max. displacement at 1.56 psi static stress. 14.6% avg. max. displacement at 4.68 psi static stress.
8. Loaded Bulk Density	0.40-1.5 lbs per cu ft	2.80 lbs per cu ft

9. Compressive Creep	Load (psi)	% Creep	Load (psi)	% Creep
	0.4	10 max.	0.44	1.45
	0.8	15 max.	0.84	2.06
	1.2	18 max.	1.28	3.65

10. Compressive Set	Load (psi)	% Creep	Load (psi)	% Creep
	0.4	5 max.	0.44	1.45
	0.8	10 max.	0.84	2.06
	1.2	13 max.	1.28	3.65
			No recovery within 24 hour test period	

66

TABLE 18   FEDERAL SPECIFICATION
PPP-C-1683 TEST SUMMARY (Continued)

NAME OF TEST	PPP-C-1683 REQUIREMENT	TEST RESULT
11. Flammability	Not less than 50% of original weight retained	99.7% of original weight retained
12. Electrostatic	Nonstatic	Nonstatic
13. Workmanship	Clean, free of foreign matter and imperfections. Less than 10% deformed or fragmented particles.	Clean, dustless, no visible foreign matter, imperfections or fragmented particles.

When comparing the results obtained in Phase II to the requirements of PPP-C-1683 (Table XVIII), it is evident that the hydroxypropyl cellulose foam exceeds the allowable property values for high peak acceleration and loaded bulk density. This departure from the standard property requirements for foams currently in use for cushioning purposes can be directly attributed to the relatively high density (5.19 lb/cu ft) of the individual pieces of Klucel foam.

The effects of the 31 day, long term humidity exposure was damaging. The amount of moisture absorbed caused material deterioration which completely altered the pnysical properties and appearance of the specimens. However, from a water solubility standpoint, hydroxypropyl cellulose foam appears promising as a loose-fill packaging material which could provide an acceptable ecological solution to a disposal problem.

The present cost of Klucel Grade J resin is $2.18/lb. This compares unfavorably with the price of polyethylene and polystyrene polymers used to produce the conventional foamed loose-fill packaging materials presently on the market. The current prices of polyethylene and polystyrene are $0.25-$0.30/lb and $0.305-$0.33/lb respectively (Reference 9). The effect of this price differential is even more noticeable when it is realized that the density of the current loose-fill foam products is approximately one-half that of the product produced in this project.

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